



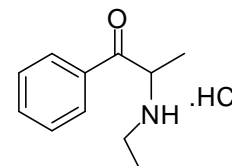
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D938b: (\pm)-N-Ethylcathinone hydrochloride

Report ID: D938b.2022.01

Chemical Formula: $C_{11}H_{15}NO.HCl$

Molecular Weight: 213.7 g/mol (HCl), 177.2 (base)



Property value

Batch No.	CAS No.	Purity estimate
10-D-14	51553-17-4 (HCl) 18259-37-5 (base)	99.7 \pm 0.4%

IUPAC name: 2-(Ethylamino)-1-phenyl-1-propanone hydrochloride

Expiration of certification: The property values are valid till 27 July 2027, 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, 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: At the recommended storage conditions this material has demonstrated stability for a period of ten years, which is captured in the stated measurement uncertainty.

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 with UV detection 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.
16 August 2022

This report supersedes any issued prior to 16 August 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. Impurities of related structure were assessed by GC-FID and HPLC with UV detection. The purity estimate 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 quantitative NMR, qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890N or 8890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 160 °C, 30 °C/min to 300 °C (3 min)
	Injector:	180 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.4%, s = 0.06% (5 sub samples in duplicate, August 2014)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, September 2015)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, July 2022)
HPLC:	Instrument:	Shimadzu Model LC-20AB Binary pump, SIL-20A HT auto sampler
	Column:	Ascentis C-18, 2.7 μm (4.6 mm x 150 mm)
	Column oven:	Ambient
	Mobile Phase:	Acetonitrile/Milli-Q water (20:80)
		0.05% TFA was present in both aqueous and organic phases
	Flow rate:	0.4 mL/min
	Detector:	Shimadzu PDASPD-M20A operating at Max Plot/240 nm
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.02% (10 sub samples in duplicate, October 2010)
	Re-analysis:	Mean = 100.0%, s = 0.0% (5 sub samples in duplicate, October 2011)
Karl Fischer analysis:	Moisture content ≤ 0.2% mass fraction (October 2010, 2011, July 2014, August 2015 & July 2022)	
Thermogravimetric analysis:	Non-volatile residue < 0.2 % mass fraction (November 2010). The volatile content (e.g. organic solvents and/or water) could not be analysed accurately because of the inherent volatility of the material.	
QNMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	600 MHz
	Solvent:	D ₂ O (4.71 ppm)
	Internal standard:	Potassium hydrogen maleate (98.6% mass fraction)
	Initial analysis:	Mean = 99.8%, s = 0.41% (5 sub samples, October 2010)

Spectroscopic and other characterisation data

ESI-MS:	Instrument: Operation: Ionisation: EM voltage: Cone voltage: Peak:	Micromass Quatro LC Micro Positive ion mode, direct infusion at 10 µL/min ESI spray voltage at 3.5 kV positive ion 650 V 21 V 178.2 (M+H ⁺) <i>m/z</i>
HS-GC-MS:	Instrument: Column: Program: Injector: Split ratio: Transfer line temp: Carrier: Solvents detected:	Agilent 6890/5973/G1888 DB-624, 30 m x 0.25 mm I.D. x 1.4 µm 50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min) 150 °C 50/1 280 °C Helium, 1.2 mL/min Ethanol and diethyl ether
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Methanol/NH ₃ (100:1.5) Single spot observed, R _f = 0.6. Visualisation with UV at 254 nm.
IR:	Instrument: Range: Peaks:	Biorad FTS300MX FT-IR 4000-400 cm ⁻¹ , KBr powder 3368, 2936, 2802, 2693, 2482, 2380, 1969, 1695, 1597, 1459, 1440, 1315, 1239, 979, 794, 699 cm ⁻¹
¹ H NMR:	Instrument: Field strength: Solvent: Spectral data:	Bruker Avance DMX-600 600 MHz D ₂ O (4.79 ppm) δ 1.38 (3H, t, <i>J</i> = 7.3 Hz), 1.61 (3H, d, <i>J</i> = 7.3 Hz), 3.16 (1H, qd, <i>J</i> = 7.3, 12.4 Hz), 3.25 (1H, qd, <i>J</i> = 7.3, 12.4 Hz), 5.17 (1H, q, <i>J</i> = 7.3 Hz), 7.63 (2H, dd, <i>J</i> = 7.8, 8.0 Hz), 7.79 (1H, dd, <i>J</i> = 7.5, 7.5 Hz), 8.03 (2H, dd, <i>J</i> = 8.2, 8.5 Hz) ppm Ethanol estimated at 0.04% mass fraction was observed in the ¹ H NMR, diethyl ether was not observed.
¹³ C NMR:	Instrument: Field strength: Solvent: Spectral data:	Bruker Avance-600 150 MHz D ₂ O δ 10.8, 15.6, 41.4, 58.1, 128.9, 129.3, 132.3, 135.5, 197.6 ppm
Melting point:		180-186 °C
Microanalysis:	Found: Calculated:	C = 61.8%; H = 7.7%; N = 6.4%; Cl = 16.8% (October, 2010) C = 61.8%; H = 7.6%; N = 6.6%; Cl = 16.6% (Calculated for C ₁₁ H ₁₅ NO.HCl)