



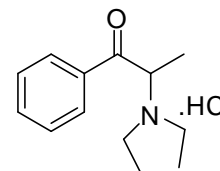
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D957: (\pm)-N,N-Diethylcathinone hydrochloride

Report ID: D957.2022.02 (Bottled 160823)

Chemical Formula: C₁₃H₂₀ClNO

Molecular Weight: 241.8 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
10-D-10	134-80-5	97.2 \pm 0.9%

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

IUPAC: 2-(Diethylamino)-1-phenyl-1-propanone hydrochloride

Expiration of certification: The property values are valid till 9 February 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: 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 4 °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 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 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.

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.
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.

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, HPLC with UV detection, qNMR, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR spectroscopy. The purity value is calculated as per Equation 1.

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

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: Agilent 7890A/6890 Column: HP-5, 30 m × 0.32 mm I.D. × 0.25 μm Program: 100 °C (1 min), 10 °C/min to 180 °C, 30 °C/min to 300 °C (3 min) Injector: 250 °C Detector Temp: 320 °C Carrier: Helium Split ratio: 20/1 Relative mass fraction of main component as the free base: Initial analysis: Mean = 99.5%, s = 0.08% (5 sub samples in duplicate, August 2012) Re-analysis: Mean = 99.7%, s = 0.04% (5 sub samples in duplicate, June 2013) Re-analysis: Mean = 99.7%, s = 0.03% (5 sub samples in duplicate, April 2016) Re-analysis: Mean = 99.7%, s = 0.01% (5 sub samples in duplicate, March 2019) Re-analysis: Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, February 2022)
HPLC:	Instrument: Shimadzu Model LC-20AB Binary pump, SIL-20A HT autosampler or Waters Binary pump 1525, 717 plus autosampler Column: X-Bridge C-18, 5 μm (4.6 mm × 150 mm) (40 °C) Mobile Phase: Acetonitrile/Milli-Q water NH ₄ OAc buffer (60:40) The aqueous phase was buffered at pH 10.8 NH ₄ OAc Flow rate: 1.0 mL/min Detector: Shimadzu PDASPD-M20A or Waters PDA-2998 operating at 240 nm Relative mass fraction of main component: Initial analysis: Mean = 99.9%, s = 0.01% (10 sub samples in duplicate, August 2010) Re-analysis: Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, August 2011)
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.
Karl Fischer analysis:	Moisture content 0.3% mass fraction (September 2010) Moisture content 0.6% mass fraction (June 2013) Moisture content 0.8% mass fraction (May 2014) Moisture content 1.1% mass fraction (March 2019) Moisture content 1.0% mass fraction (January 2022)
qNMR:	Instrument: Bruker Avance-400 Field strength: 400 MHz Solvent: D ₂ O (4.79 ppm) Internal standard: Potassium hydrogen maleate (98.8% mass fraction) Initial analysis: Mean = 97.3%, s = 0.25% (5 sub samples, August 2010)

Spectroscopic and other characterisation data

LC/ESI -MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 μ L/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	206.1 (M+H ⁺) <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 μ m
	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:	Ethanol, diethyl ether and ethyl acetate
TLC:	Conditions:	Kieselgel 60F254. Methanol/NH ₃ (100/1.5) Single spot observed, R _f = 0.7. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1} , KBr powder
	Peaks:	3353, 2983, 2943, 2616, 1687, 1447, 1390, 1290, 1233, 1129, 1015, 976, 707 cm^{-1}
¹ H NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	600 MHz
	Solvent:	D ₂ O (4.79 ppm)
	Spectral data:	δ 1.37 (3H, t, <i>J</i> = 7.0 Hz), 1.45 (3H, t, <i>J</i> = 7.0 Hz), 1.64 (3H, d, <i>J</i> = 7.1 Hz), 3.20 (1H, m), 3.37 (2H, m), 3.54 (1H, m), 5.30 (1H, q, <i>J</i> = 7.3 Hz), 7.65 (2H, t, <i>J</i> = 7.8 Hz), 7.82 (2H, t, <i>J</i> = 7.5 Hz), 8.07 (2H, t, <i>J</i> = 7.5 Hz) ppm Ethanol and diethylamine hydrochloride estimated at 0.4% and 1.1% mass fraction respectively were observed in the ¹ H NMR.
¹³ C NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	151 MHz
	Solvent:	D ₂ O
	Spectral data:	δ 7.0, 9.1, 13.7, 44.0, 47.2, 62.2, 128.5, 128.9, 132.2, 135.2, 197.1 ppm
Melting point:		154-164 °C
Microanalysis:	Found:	C = 64.2%; H = 8.5%; N = 5.9%; Cl = 14.8% (August, 2010)
	Calculated:	C = 64.6%; H = 8.3%; N = 5.8%; Cl = 14.7% (Calculated for C ₁₃ H ₂₀ ClNO)