



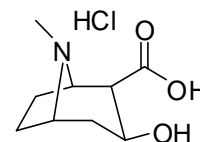
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D746b: Ecgonine hydrochloride

Report ID: D746b.2019.03

Chemical Formula: $C_9H_{15}NO_3 \cdot HCl$

Molecular Weight: 221.7 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
08-D-09	5796-31-6	99.3 ± 0.7%

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

IUPAC name: (1*R*,2*R*,3*S*,5*S*)-2-Carboxy-3-hydroxy-8-methyl-8-azoniabicyclo[3.2.1]octane chloride

Expiration of certification: The property values are valid till 3 September 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: 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. 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 annual stability trials. The long-term stability of the compound in solution has not been examined.

Safety: Treat as 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

Product release date: 11 November 2008

measurement.gov.au

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 quantitative nuclear magnetic resonance (qNMR). The three-proton singlet at 2.8 ppm was measured against a certified internal standard of maleic acid.

Supporting evidence is provided by Karl Fischer analysis, ¹H NMR analysis and elemental microanalysis.

QNMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz
	Solvent:	D ₂ O
	Internal standard:	Potassium hydrogen maleate
	Purity estimate:	99.3% (mass fraction %, mean of four samples, s = 0.16 %, August 2008)
QNMR:	Instrument:	Bruker DMX-600
	Field strength:	400 MHz
	Solvent:	D ₂ O
	Internal standard:	Potassium hydrogen maleate
	Purity estimate:	99.5% (mass fraction %, mean of five samples, s = 0.79 %, September 2009)
QNMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	D ₂ O
	Internal standard:	Maleic acid
	Purity estimate:	99.1% (mass fraction %, mean of five samples in duplicate, s = 0.28 %, January 2011)
QNMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	D ₂ O
	Internal standard:	Maleic acid
	Purity estimate:	99.1% (mass fraction %, mean of five samples in duplicate, s = 0.32 %, November 2013)
QNMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	D ₂ O
	Internal standard:	Maleic acid
	Purity estimate:	99.7% (mass fraction %, mean of five samples in duplicate, s = 0.21 %, October 2016)
Karl Fischer analysis:		Moisture content 0.3% mass fraction (September 2008)
		Moisture content 0.2% mass fraction (January 2011 and November 2013)
		Moisture content ≤ 0.1% mass fraction (November 2016 and September 2019)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	DB-1701, 30 m × 0.25 mm I.D. × 0.25 μm
	Program:	180 °C (1 min), 4 °C/min to 200 °C, 6 °C/min to 275 °C (6.5 min)
	Injector:	230 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.3 mL/min
	Split ratio:	splitless
	The retention time of the <i>bis</i> -trimethylsilyl derivative 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.	
	4.3 min:	329 (M ⁺ , 9), 314 (15), 212 (11), 147 (12), 96 (62), 83 (100), 73 (39) <i>m/z</i>
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Methanol/conc. NH ₃ (200/3) Single spot observed, R _f = 0.27. Visualisation with ninhydrin
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm ⁻¹ , KBr powder
	Peaks:	3267, 3091, 2964, 1709, 1431, 1351, 1227, 1140, 1041, 881, 607 cm ⁻¹
¹ H NMR:	Instrument:	Bruker DMX600
	Field strength:	600 MHz
	Solvent:	D ₂ O (4.79)
	Spectral data:	δ 1.98-2.19 (4H, m), 2.25-2.41 (2H, m), 2.78 (3H, s), 3.18 (1H, dd, <i>J</i> = 2.3, 7.1 Hz), 3.92 (1H, m), 4.10 (1H, d, <i>J</i> = 7.3 Hz), 4.41 (1H, m) ppm
¹³ C NMR:	Instrument:	Bruker DMX600
	Field strength:	150 MHz
	Solvent:	D ₂ O
	Spectral data:	δ 23.2, 24.0, 35.5, 38.9, 49.5, 60.8, 63.6, 64.6, 176.4 ppm
Melting point:		233 °C
Microanalysis:	Found:	C = 49.1 %; H = 7.4 %; N = 6.3 % (August 2008)
	Calculated:	C = 48.8 %; H = 7.3 %; N = 6.3 % (Calculated for C ₉ H ₁₅ NO ₃ .HCl)