Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

NATA
ACCREDITED FOR TECHNICAL COMPETENCE

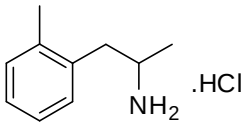
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D912b: Ortetamine hydrochloride

Report ID: D912b.2020.01

Chemical Formula: $C_{10}H_{15}N.HCl$

Molecular Weight: 185.7 g/mol (HCl), 149.2 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
13-D-29	77083-24-0 (HCl) 5580-32-5 (base)	97.0%

IUPAC name: 1-(2-Methylphenyl)-2-propanamine hydrochloride

Expiration of certification: The property values are valid till 30 April 2025, 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 reference material is recommended for qualitative analysis only.

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 ºC in a closed container in a dry, dark area.

Stability: This material has demonstrated stability over a minimum period of three years. 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.
14 May 2020

This report supersedes any issued prior to 14 May 2020

NATA logo notice: Accredited for compliance with ISO 17034. Accreditation No. 198 / Corporate Site No. 20844. The results of the tests, calibrations and/or measurements included in this document are traceable to Australian/national standards.

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. The purity estimate was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, Karl Fischer analysis and ^{1}H NMR spectroscopy. The purity value is calculated as per Equation 1.

$$\text{Purity} = (100\ \% - I_{ORG}) \times (100\ \% - I_{VOL} - I_{NVR}) \qquad \text{Equation 1}$$

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:	Agilent 6890, Agilent 7890
	Column:	HP-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 150 °C, 30 °C/min to 300 °C (3 min)
	Injector:	220 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component as the free base:	
	Initial analysis:	Mean = 99.8%, s = 0.05% (10 sub samples in duplicate, June 2014)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, June 2015)
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, June 2016)
	Re-analysis:	Mean = 99.7%, s = 0.01% (5 sub samples in duplicate, May 2017)
	Re-analysis:	Mean = 99.9%, s = 0.004% (5 sub samples in duplicate, April 2020)
Karl Fischer analysis:		Moisture content 2.3% mass fraction (June 2014) Moisture content 3.0% mass fraction (June 2015) Moisture content 2.2% mass fraction (June 2016) Moisture content 2.3% mass fraction (May 2017) Moisture content 2.2% mass fraction (November 2019)

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 (1 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	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 (8.6 min):	134 (4), 117 (6), 115 (6), 105 (18), 91 (20), 77 (16), 44 (100) *m/z*
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 μL/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	10 V
	Peak:	150.0 ($M+H^+$) *m/z*
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
	Split ratio:	50/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Solvents detected:	Isopropanol, diethyl ether
TLC:	Conditions:	Kieselgel $60F_{254}$. Methanol/concentrated aqueous ammonia (200/3) Single spot observed, R_f = 0.31. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3023, 2935, 2589, 2494, 1594, 1504, 1488, 1391, 1203, 1139, 1074, 748 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	MeOH-d_4 (3.31 ppm)
	Spectral data:	δ 1.26 (3H, d, *J* = 6.6 Hz), 2.36 (3H, s), 2.84 (1H, dd, *J* = 9, 13.6 Hz), 3.05 (1H, dd, *J* = 5.9, 13.6 Hz), 3.52 (1H, m), 7.15-7.20 (4H, m) ppm Isopropanol estimated at 0.1% and diethyl ether at 0.1% mass fraction was observed in the ^{1}H NMR.
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	MeOH-d_4 (49.0 ppm)
	Spectral data:	δ 18.3, 19.6, 39.1, 49.2, 127.4, 128.5, 131.2, 131.8, 135.6, 137.7 ppm
Melting point:		177-178 °C
Microanalysis:	Found:	C = 63.4%; H = 8.7%; N = 7.4%, Cl = 18.9% (June, 2014)
	Calculated:	C = 63.2%; H = 8.7%; N = 7.4%, Cl = 18.7 (Calculated for $C_{10}H_{15}N.HCl$ + 2.3% water)