

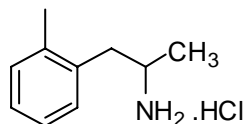


CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

Report ID: D912.2012.01

Compound Name: **Ortetamine hydrochloride**
Collection No: D912
Chemical Formula: C₁₀H₁₅N.HCl
CAS No: 5580-32-5 (base)
Structure:

Description: White solid
Batch No: 06-D-09
Molecular Weight: 185.7 (HCl), 149.2 (base)
Release date: 30th April 2007



Synonyms: α -2-Dimethylbenzene-ethanamine; *o*- α -dimethyl-phenethylamine; *o*-methyl- α -methylphenylethylamine; 2-methylamphetamine; DL-*o*- α -dimethylphenethylamine; ortetamin; ortetamine; *o*- α -dimethylphenethylamine

Purity (mass fraction): 97.2 \pm 1.5 % (95 % confidence interval)

Purity estimate obtained by quantitative nuclear magnetic resonance (QNMR) using the three proton multiplet at 2.4 ppm against a certified internal standard of potassium hydrogen maleate. Supporting evidence provided by GC-FID, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR.

QNMR: Instrument: Bruker DMX-600
Field strength: 600 MHz Solvent: D₂O (4.79 ppm)
Internal standard: Potassium hydrogen maleate (98.8% mass fraction)
Initial analysis: Mean (2.4 ppm) = 97.2%, s = 0.65% (5 sub samples, April 2007)

GC-FID: Instrument: Varian CP3800
Column: VF-1ms, 29.8 m \times 0.32 mm I.D. \times 0.25 μ m
Program: 60 $^{\circ}$ C (1 min), 10 $^{\circ}$ C/min to 150 $^{\circ}$ C, 30 $^{\circ}$ C/min to 300 $^{\circ}$ C (3 min)
Injector: 220 $^{\circ}$ C Detector Temp: 320 $^{\circ}$ C
Carrier: Helium Split ratio: 20/1
Relative peak area response of main component:
Initial analysis: Mean = 99.3%, s = 0.03% (7 sub samples in duplicate, January 2010)

GC-FID: Instrument: Agilent 6890N/7890
Column: HP-1, 29.5 m \times 0.32 mm I.D. \times 0.25 μ m
Program: 60 $^{\circ}$ C (1 min), 10 $^{\circ}$ C/min to 150 $^{\circ}$ C, 30 $^{\circ}$ C/min to 300 $^{\circ}$ C (3 min)
Injector: 220 $^{\circ}$ C Detector Temp: 320 $^{\circ}$ C
Carrier: Helium Split ratio: 20/1
Relative peak area response of main component:
Initial analysis: Mean = 99.2%, s = 0.03% (10 sub samples in duplicate, March 2007)
Re-analysis: Mean = 99.2%, s = 0.01% (5 sub samples in duplicate, December 2008)
Re-analysis: Mean = 99.4%, s = 0.02% (7 sub samples in duplicate, November 2012)

Thermogravimetric analysis: Volatile content 0.53% mass fraction (2 sub samples, December 2006)
Non volatile content not determined due to nature of the material.

Karl Fischer analysis: Moisture content < 2.0% mass fraction. (January 2007 - December 2009)
Moisture content 1.4% mass fraction. (November 2012)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890 / 5973	
	Column:	DB-35MS, 30 m × 0.25 mm I.D. × 0.25 µm	
	Program:	90 °C (1 min), 8 °C/min to 300 °C, (10 min)	
	Injector:	250°C	Transfer line temp: 310 °C
	Carrier:	Helium, 1.0 mL/min	Split ratio: splitless
	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.		
	7.5 min: 134 (2), 117 (2), 115 (2), 105 (6), 91 (7), 77 (6), 44 (100) m/z		
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . conc. NH ₃ / methanol (1.5/100) Single spot observed, R _f = 040. Visualisation with UV at 254 nm	
IR:	Instrument:	Biorad FTS300MX FT-IR	
	Range:	4000-400cm ⁻¹ , KBr pellet	
	Peaks:	3023, 2935, 2589, 2494, 1594, 1504, 1488, 1391, 1203, 1139, 1074, 748 cm ⁻¹	
¹ H NMR:	Instrument:	Bruker DMX-500	
	Field strength:	500 MHz	Solvent: CD ₃ OD (3.30 ppm)
	Spectral data:	δ 1.25 (3H, d, <i>J</i> = 6.6 Hz), 2.35 (3H, s), 2.83 (1H, dd, <i>J</i> = 9, 13.6 Hz), 3.06 (1H, dd, <i>J</i> = 5.9, 13.6 Hz), 3.51 (1H, m), 7.15-7.20 (4H, m) ppm	
	Isopropanol estimated at 0.25% mass fraction was observed in the ¹ H NMR		
¹³ C NMR:	Instrument:	Bruker DMX-500	
	Field strength:	126 MHz	Solvent: CD ₃ OD (49.0 ppm)
	Spectral data:	δ 18.3, 19.6, 39.0, 49.3, 127.4, 128.5, 131.2, 131.8, 135.6, 137.7 ppm	
Melting point:	167.4-169.4°C		
Microanalysis:	Found: C = 62.2 %; H = 8.9 %; N = 7.9% (December 2006) Calc: C = 64.7 %; H = 8.7 %; N = 7.5% (Calculated for C ₁₀ H ₁₅ N.HCl)		

The Synthesis and Certification of this Reference Material is supported by the Australian Government through the *Anti-Doping Research Program (ADRP)* of the Department of Communications, Information Technology and the Arts.

Expiration of certification

The property values are valid till 27th November 2017, 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.

The long-term stability of the compound in solution has not been examined.

This material has demonstrated stability over a minimum period of 3 years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials.

Homogeneity assessment

The homogeneity of the material was assessed using purity assay by GC-FID on 5 randomly selected 1-2 mg samples of the material. The material was judged to be 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.

Metrological Traceability

The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. The purity was derived by subtraction of the mass of impurities from the mass of the reference material. Organic purity is traceable to the SI-derived coherent unit one through chromatographic separation and response factor determination of individual components. Volatile and non-volatile residue content is directly traceable to mass through use of Karl Fischer and thermogravimetric analysis. 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).

Recommended storage

When not in use this material should be stored at or below 20 °C in a closed container in a dry, dark area.

Intended Use

For *in vitro* laboratory analysis only.

Caution

Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice

Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 11 January, 2013.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 8th January 2013.



This document is issued in accordance with NATA's accreditation requirements.

Accredited for compliance with ISO Guide 34.

This document shall not be reproduced except in full.

Accreditation Number : 198

Corporate Site Number : 14214

