Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

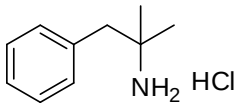
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D781: Phentermine hydrochloride

Report ID: D781.2021.02

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

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



Certified value

Batch No.	CAS No.	Purity (mass fraction)
08-D-16	1197-21-3 (HCl) 122-09-8 (base)	97.4 ± 0.4%

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

IUPAC name: 2-Methyl-1-phenyl-2-propanamine hydrochloride.

Expiration of certification: The property values are valid till 10 September 2031, i.e. ten 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 sourced from an external supplier, 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 ºC 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 ten years. The measurement uncertainty at the 95% confidence 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.

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.
15 September 2022

This report supersedes any issued prior to 15 September 2022.

NATA Accreditation No. 198 / Corporate Site No. 14214.

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 six-proton doublet of doublets at 1.3 ppm was measured against a certified internal standard of potassium hydrogen maleate.

Supporting evidence is provided by GC-FID, thermogravimetric analysis, Karl Fischer analysis, ^{1}H NMR spectroscopy and elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890N/8890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 8 °C/min to 120 °C, 30 °C /40 °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:	
	Initial analysis:	Mean = 99.9%, s = 0.02% (5 sub samples in duplicate, December 2011)
	Re-analysis:	Mean = 99.8%, s = 0.07% (5 sub samples in duplicate, November 2016)
	Re-analysis:	Mean = 100.0%, s = 0.002% (5 sub samples in duplicate, September 2021)
GC-FID:	Instrument:	Varian CP-3800
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 6 °C/min to 120 °C, 20 °C/min to 300 °C (5 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of main component:	
	Initial analysis:	Mean = 99.9%, s = 0.02% (10 sub samples in duplicate, November 2008)
Karl Fischer analysis:		Moisture content ca. 2.2% mass fraction (November 2008, 2011 and September 2016) Moisture content ca. 2.0% mass fraction (August 2021)
Thermogravimetric analysis:		Volatile content not determined due to the nature of the material
QNMR:	Instrument:	Bruker Gyro-300
	Field strength:	300 MHz
	Solvent:	D_2O
	Internal standard:	Potassium hydrogen maleate (98.8% mass fraction)
	Initial analysis:	Mean (1.3 ppm) = 97.4%, s = 1.1% (5 sub samples, December 2008)

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Micromass Quatro Micro
	Operation:	Positive ion mode, direct infusion at 5 µL/min
	Ionisation:	ESI spray voltage at 3.0 kV positive ion
	EM voltage:	500 V
	Cone voltage:	20 V
	Peak:	150.0 ($M+H^+$) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Diisopropylether/diethyether/diethylamine (45:45:10) Single spot observed, R_f = 0.57. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm^{-1}, KBr powder
	Peaks:	3536, 2983, 2880, 2778, 2661, 2575, 2358, 2076, 1608, 1493, 1458, 1373, 1283, 1172 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Gyro-300
	Field strength:	300 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 1.36 (6H, s), 2.96 (2H, s), 7.29-7.44 (5H, m) ppm
^{13}C NMR:	Instrument:	Bruker Gyro-300
	Field strength:	75 MHz
	Solvent:	D_2O
	Spectral data:	δ 25.0, 45.6, 55.1, 127.9, 129.1, 131.0, 135.2 ppm
Melting point:		198-200 °C
Microanalysis:	Found:	C = 63.4%; H = 8.7%; N = 7.5%
	Calculated:	C = 64.7%; H = 8.8%; N = 7.4% (Calc for $C_{10}H_{15}N.HCl$)
	Calculated:	C = 63.5%; H = 8.7%; N = 7.4% (Calc for $C_{10}H_{15}N.HCl$ containing 0.2% H_2O)