Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

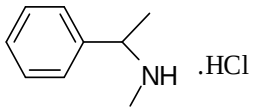
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D525: N-Methyl-1-phenylethylamine hydrochloride

Report ID: D525.2020.03

Chemical Formula: $C_9H_{13}N.HCl$

Molecular Weight: 171.7 g/mol (HCl salt), 135.2 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
98-00302	**32512-24-6**	**99.7 ± 0.4%**

Synonyms: N,α–dimethyl-benzenemethanamine hydrochloride
N-methyl-alpha-phenylethylamine hydrochloride

Expiration of certification: The property values are valid till 29 November 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 crystals prepared by synthesis or sourced from an external supplier, 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 five 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 seven randomly selected 1-2 mg sub 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.

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

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
13 October 2022.

This report supersedes any issued prior to 14 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 purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR spectroscopy. The purity value is calculated as per Equation 1.

Purity = (100% - I_{ORG}) x (100% - I_{VOL} – I_{NVR}) Equation 1

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue

Supporting evidence is provided by elemental microanalysis.

GC-FID:	Instrument:	HP5890
	Column:	Zebron ZB-1 Capillary, 30 m x 0.32 mm I. D. x 0.25 μm
	Program:	180 °C (1 min), 10 °C/min to 250 °C
	Injector Temp:	250 °C
	Detector Temp:	325 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.1% (7 sub samples, February 1998)
	Re-analysis:	Mean = 99.8%, s = 0.003% (5 sub samples in duplicate, July 2005)
GC-FID:	Instrument:	Varian CP3800
	Column:	HP-5 Capillary, 30 m x 0.32 mm I. D. x 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 120 °C, 30 °C/min to 300 °C (3 min)
	Injector Temp:	250 °C
	Detector Temp:	325 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 100%, s = 0% (5 sub samples, July 2010)
	Re-analysis:	Mean = 100%, s = 0.007% (5 sub samples in duplicate, July 2015)
	Re-analysis:	Mean = 100%, s = 0.007% (5 sub samples in duplicate, October 2020)
Karl Fischer:		Moisture content 0.2% mass fraction (August 2006)
		Moisture content 0.2% mass fraction (June 2010)
		Moisture content < 0.2% mass fraction (June 2015)
		Moisture content < 0.1% mass fraction (November 2020)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP5890/5970B
	Column:	HP Ultra-2, 12m x 0.22 mm I.D. Film thickness 0.11μm
	Program:	60 °C to 300 °C at 10 °C/min
	Injector:	230 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 ml/min
	Split ratio:	10/1
	The retention times 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.	
	Parent (5.0 min):	134, 120, 105, 91, 77, 65, 58, 51, 42 *m/z*
HPLC:	Method:	Peak area percentage of total: 100% (3 samples, March 1998)
	Column:	Alltima C-18, 5 μm (4.6 mm x 150 mm)
	Mobile Phase:	Methanol/water/diethylamine (60:40:0.5), pH 8.4
	Flow Rate:	0.8 ml/min
	Detector:	Refractive Index
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400cm^{-1}. KBr pellet
	Peaks:	2965, 2714, 2473, 1589, 1499, 1476, 1458, 1419, 1387, 1222, 1058, 1030, 909, 766, 703, 548 cm^{-1}
TLC:	Conditions:	Kieselgel 60F$_{254}$. Cyclohexane/toluene/diethylamine (75:15:10) Single spot observed, R_f = 0.32 (UV detection)
^{1}H NMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz
	Solvent:	MeOH-d_4
	Peaks:	δ 1.68 (d ,3H, *J* = 6.8 Hz), 2.56 (s, 3H), 4.34 (q, 1H, *J* = 6.8 Hz), 7.44-7.51 (m, 5H) ppm
^{13}C NMR:	Instrument:	Bruker DMX-600
	Field strength:	151 MHz
	Solvent:	MeOH-d_4
	Peaks:	δ 19.2, 31.5, 60.5, 128.7, 130.5, 130.7, 137.5 ppm.
Microanalysis:	Found:	C = 63.3%, H = 8.3%, N = 8.2%
	Calculated:	C = 63.0%, H = 8.2%, N = 8.2%
Melting point:		166-171 °C