Australian Government
Department of Industry, Innovation and Science

National Measurement Institute

NATA ACCREDITED FOR TECHNICAL COMPETENCE

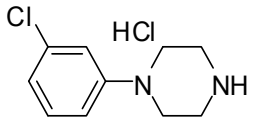
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D907: 1-(3-Chlorophenyl)piperazine hydrochloride

Report ID: D907.2017.02

Chemical Formula: $C_{10}H_{14}Cl_2N_2$

Molecular Weight: 233.1 g/mol (HCl), 196.7 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
06-D-04	13078-15-4 (HCl) 6640-24-0 (base)	99.6 %

IUPAC name: 1-(3-Chlorophenyl)piperazine dihydrochloride

Expiration of certification: The property values are valid till 23 February 2022, 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: Off-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 reference material should be used 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 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.

Homogeneity assessment: The homogeneity of the material was assessed using purity assay by GC-FID on nine 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.

Caution: 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.
11 February 2020

This report supersedes any issued prior to 11 February 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: Neither NMIA as a representative of the Commonwealth of Australia, nor any person acting on NMIA'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 document.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified 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:	Agilent 6890N
	Column:	HP-1, 30 m x 0.32 mm I.D. x 0.25 µm
	Program:	100 °C (1 min), 15 °C/min to 150 °C (6 min), 30 °C/min to 310 °C (8 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area response of main component:	
	Initial analysis:	Mean = 99.8%, s = 0.02% (9 sub samples in duplicate, June 2006)
	Re-analysis:	Mean = 99.7%, s = 0.03% (5 sub samples in duplicate, February 2007)
	Re-analysis:	Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, February 2008)
	Re-analysis:	Mean = 99.6%, s = 0.01% (5 sub samples in duplicate, February 2009)
	Re-analysis:	Mean = 99.8%, s = 0.04% (5 sub samples in duplicate, February 2012)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, February 2017)
Thermogravimetric analysis:		Initial volatile content < 0.1% (June 2006 and June 2007) Non-volatile residue not determined
Karl Fischer analysis:		Moisture content < 0.3% mass fraction (February 2008, February 2009, February 2012 and February 2017)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP 5890/5971A
	Column:	ZB-5, 26 m × 0.25 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 250 °C (2 min)
	Injector:	220°C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	20/1
	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 (14.4 min):	198 (M^+, 9), 196 (M^+, 25), 156 (32), 154 (100), 138 (13), 111 (15), 75 (14), 56 (22) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Dichloromethane/methanol/conc. ammonia (90/10/0.6) Single spot observed, R_f = 0.49. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm^{-1}, KBr powder
	Peaks:	3200-2700 (broad), 2484, 1593, 1489, 1418, 1257, 1157, 1102, 943, 751, 676 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX500
	Field strength:	500 MHz
	Solvent:	MeOH-d_4 (3.31 ppm)
	Spectral data:	δ 3.38-3.40 (4H, m), 3.46-3.48 (4H, m), 6.92 (1H, dd, *J* = 1.2, 7.9 Hz), 6.97 (1H, dd, *J* = 2.0, 8.2 Hz), 7.05 (1H, dd, *J* = 2.0, 2.0 Hz), 7.27 (1H, dd, *J* = 8.2, 8.2 Hz) ppm
^{13}C NMR:	Instrument:	Gyro 300
	Field strength:	75 MHz
	Solvent:	MeOH-d_4 (49.0 ppm)
	Spectral data:	δ 45.4, 48.1, 116.9, 118.5, 122.5, 132.4, 136.9, 153.7 ppm
Melting point:		212-213 °C
Microanalysis:	Found:	C = 51.8 %, H = 6.0 %; N = 12.1% (March 2006)
	Calculated:	C = 51.5 %, H = 6.1 %; N = 12.0% (Calculated for $C_{10}H_{14}Cl_2N_2$)