Australian Government
Department of Industry, Science and Resources

National Measurement Institute

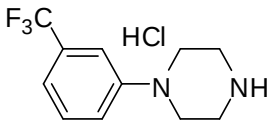
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D906: 1-(3-Trifluoromethylphenyl)piperazine hydrochloride

Report ID: D906.2018.04 (Bottled 161027)

Chemical Formula: $C_{11}H_{13}F_3N_2.HCl$

Molecular Weight: 266.7 g/mol (HCl), 230.2 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
06-D-03	16015-69-3 (HCl) 15532-75-9 (base)	99.8 ± 0.3%

IUPAC name: 1-[3-(Trifluoromethyl)phenyl]piperazine hydrochloride

Expiration of certification: The property values are valid till 5 November 2023, 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 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.

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.
19 September 2022

This report supersedes any issued prior to 19 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 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, 29.50 m x 0.32 mm I.D. x 0.25 µm
	Program:	100 °C (1 min), 10 °C/min to 160 °C, 30 °C/min to 300 °C (5 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area response of main component:	
	Initial analysis:	Mean = 99.98%, s = 0.01% (10 sub samples in duplicate, June 2006)
	Re-analysis:	Mean = 99.73%, s = 0.01% (5 sub samples in duplicate, February 2009)
	Re-analysis:	Mean = 99.97%, s = 0.02% (5 sub samples in duplicate, December 2013)
	Re-analysis:	Mean = 99.92%, s = 0.02% (5 sub samples in duplicate, November 2018)
Thermogravimetric analysis:		Volatile content 0.2% mass fraction (February 2009). Non-volatile residue not determined.
Karl Fischer analysis:		Moisture content < 0.2% mass fraction (February 2009 and December 2013 and November 2018)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP 5890
	Column:	ZB-5, 26 m × 0.25 mm I.D. × 0.25 □m
	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 (11.7 min):	230 (M^+, 19), 188 (100), 172 (13), 145 (16), 56 (20) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Dichloromethane/methanol/conc. ammonia (90/10/0.6) Single spot observed, R_f = 0.54. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm^{-1}, KBr powder
	Peaks:	3200, 2940, 2724, 2481, 1614, 1590, 1454, 1354, 1309, 1173, 1108, 945, 778, 692 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX600
	Field strength:	600 MHz
	Solvent:	MeOH-d_4 (3.31 ppm)
	Spectral data:	δ 3.42-3.44 (4H, m), 3.53-3.55 (4H, m), 7.20 (1H, d, *J* = 9.1 Hz), 7.29 (1H, s), 7.30 (1H, d, *J* = 10.2 Hz), 7.48 (1H, dd, *J* = 9.1, 10.2 Hz) ppm
^{13}C NMR:	Instrument:	Gyro 300
	Field strength:	75 MHz
	Solvent:	MeOH-d_4 (49 ppm)
	Spectral data:	δ 45.5, 48.1, 114.8 (q, *J* = 3.6 Hz), 119.0 (q, *J* = 3.6 Hz), 122.0, 126.5 (q, *J* = 270.0 Hz), 132.0, 133.4 (q, *J* = 31.8 Hz), 152.9 ppm
Melting point:		237-239 ºC
Microanalysis:	Found:	C = 49.6 %, H = 5.4 %; N = 10.5% (March 2006)
	Calculated:	C = 49.5 %, H = 5.3 %; N = 10.5% (Calculated for $C_{11}H_{13}F_3N_2$.HCl)