Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

NATA
ACCREDITED FOR TECHNICAL COMPETENCE

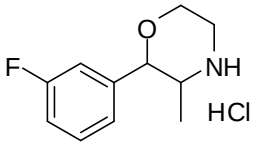
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D1061: 3-Fluorophenmetrazine hydrochloride

Report ID: D1061.2022.01

Chemical Formula: $C_{11}H_{14}FNO.HCl$

Molecular Weight: 231.7 g/mol (HCl), 195.2 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
16-D-01	**1803562-83-5 (HCl)** **1350768-28-3 (base)**	**98.3% ± 1.5%**

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

IUPAC name: 2-(3-Fluorophenyl)-3-methylmorpholine hydrochloride (1:1).

Expiration of certification: The property values are valid till 25 March 2027, 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 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 reference material is recommended for qualitative analysis only and is not intended for use as a calibrator. The material does not have certified reference material status as metrological traceability of the stated purity value to the SI unit for mass (kg) has not been established.

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% 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.
4 April 2022

This report supersedes any issued prior to 4 April 2022.

Characterisation Report:

The purity value was obtained by quantitative nuclear magnetic resonance (qNMR). The three-proton doublet at 0.96 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, headspace GC-MS analysis of occluded solvents and elemental microanalysis.

QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Internal standard:	Potassium hydrogen maleate (100 % mass fraction)
	Initial analysis:	Mean (0.98 ppm) = 98.3%, s = 0.6% (5 sub samples, January 2016)
GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	100 °C (1 min), 5 °C/min to 180 °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 the main componentas the free base:	
	Initial analysis:	Mean = 99.4%, s = 0.04% (10 sub samples in duplicate, January 2016)
	Re-analysis:	Mean = 99.4%, s = 0.02% (5 sub samples in duplicate, February 2017)
	Re-analysis:	Mean = 99.4%, s = 0.02% (5 sub samples in duplicate, March 2022)
Karl Fischer analysis:		Moisture content 0.2% mass fraction (January 2016 and February 2017) Moisture content 0.3% mass fraction (March 2022)
Thermogravimetric analysis:		Non volatile residue < 0.2% mass fraction (January 2016). The volatile content (e.g. organic solvents and/or water) could not be determined by thermogravimetric analysis

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	HP-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	100 °C (1 min), 5 °C/min to 180 °C, 20 °C/min to 300 °C (5 min)
	Injector:	250 °C,
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention time of the free base compound is reported with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Free base (10.2 min):	195 (M^+, 13), 123 (13), 95 (12), 71 (100), 56 (39), 43 (32), 42 (46) *m/z*
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 µm
	Program:	50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min)
	Injector:	150 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Split ratio:	50/1
	Solvents detected:	None
TLC:	Conditions:	Kieselgel 60F_{254}. TBME/diethyl ether/diethyl amine (19:19:2) Single spot observed, R_f = 0.33
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	2937, 2799, 2757, 2700, 2660, 2507, 2467, 1590, 1449, 1267, 1096, 1028, 937, 868, 788, 771, 691, 466 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 1.07 (3H, d, *J* = 6.7 Hz), 3.41-3.49 (2H, m), 3.54 (1H, dq, *J* = 6.6, 10.0 Hz), 4.00 (1H, m), 4.22 (1H, m), 4.55 (1H, d, *J* = 10.0 Hz), 7.17-7.26 (3H, m), 7.45 (1H, m) ppm
		Acetic acid estimated at 0.1% mass fraction was observed in the ^{1}H NMR
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	D_2O (TSP-d_4, 0.0 ppm)
	Spectral data:	δ 16.9, 46.2, 57.6, 66.7, 84.1, 117.3 (d, $J_{C\text{-}F}$ = 22.6 Hz), 119.4 (d, $J_{C\text{-}F}$ = 21.1 Hz), 126.4 (d, $J_{C\text{-}F}$ = 2.8 Hz), 133.7 (d, $J_{C\text{-}F}$ = 8.4 Hz), 141.0 (d, $J_{C\text{-}F}$ = 7.5 Hz), 165.5 (d, $J_{C\text{-}F}$ = 244.6 Hz) ppm
^{19}F NMR:	Instrument:	Bruker Avance III-500
	Field strength:	470 MHz
	Solvent:	D_2O
	Spectral data:	δ -112.9 ppm
Melting point:		231-235 °C
Microanalysis:	Found:	C = 57.1%; H = 6.6%; N = 6.1%, Cl = 15.3%, F = 8.3% (February, 2016)
	Calculated:	C = 57.0%; H = 6.5%; N = 6.1%, Cl = 15.3%, F = 8.2% (Calculated for $C_{11}H_{14}FNO.HCl$)