Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

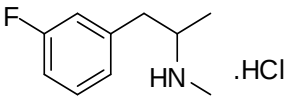
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D994: (±)-3-Fluoromethamphetamine hydrochloride

Report ID: D994.2024.01 (Bottled 160817)

Chemical Formula: $C_{10}H_{14}FN.HCl$

Molecular Weight: 203.7 g/mol



Property value

Batch No.	CAS No.	Purity by GC-FID
13-D-09	**1324717-74-9**	**99.8 ± 0.5%**

IUPAC name: 1-(3-Fluorophenyl)-N-methyl-2-propanamine hydrochloride.

Expiration of certification: The property values are valid till 18 March 2029, 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 powder prepared by synthesis 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 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 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.
23 April 2024

This report supersedes any issued prior to 23 April 2024.

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. Impurities of related structure were assessed by GC-FID. The purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID. 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 qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800 or Agilent 8890
	Column:	VF-1ms, 30 m × 0.32 mm I.D. × 0.25 μm or HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	80 °C (1 min), 5 °C/min to 120 °C, 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component as the free base:	
	Initial analysis:	Mean = 99.8%, s = 0.02% (10 sub samples in duplicate, June 2013)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, May 2014)
	Re-analysis:	Mean = 99.8%, s = 0.003% (5 sub samples in duplicate, July 2015)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, May 2016)
	Re-analysis:	Mean = 99.9%, s = 0.04% (5 sub samples in duplicate, September 2019)
	Re-analysis:	Mean = 99.8%, s = 0.005% (5 sub samples in duplicate, March 2024)
Karl Fischer analysis:		Moisture content 0.2% mass fraction (May 2013) Moisture content < 0.1% mass fraction (May 2014, April 2015 & June 2016) Moisture content 0.1% mass fraction (April 2024)
Thermogravimetric analysis:		The non-volatile residue < 0.1% mass fraction. The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material and/or degradation at elevated temperatures (June 2013).

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	The retention time of the free base 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 (8.1 min):	166 (M-1, 1), 152 (4), 135 (2), 109 (16), 83 (6), 58 (100), 42 (6) *m/z*
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 μL/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	30 V
	Peak:	168 ($M+H^+$) *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
	Split ratio:	50/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Solvents detected:	Diethyl ether, dichloromethane, isopropyl alcohol and ethyl acetate.
TLC:	Conditions:	Kieselgel $60F_{254}$. $MeOH/NH_3$ (100/1.0) Single spot observed, R_f = 0.3. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	2974, 2796, 2735, 2459, 2362, 1589, 1482, 1449, 1253, 1144, 1056, 951, 883, 788, 748, 692, 455 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance -400
	Field strength:	400 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 1.26 (3H, d, *J* = 6.6 Hz), 2.69 (3H, s), 2.89 (1H, dd, *J* = 8.0, 13.8 Hz), 3.07 (1H, dd, *J* = 6.2, 13.9 Hz), 3.54 (1H, m), 7.05-7.12 (3H, m), 7.36-7.42 (1H, m) ppm
	Isopropanol (0.03%), diethyl ether (0.02%) and ethyl acetate (0.02%) estimated mass fraction was observed in the ^{1}H NMR.	
^{13}C NMR:	Instrument:	Bruker Avance -400
	Field strength:	100 MHz
	Solvent:	D_2O
	Spectral data:	δ 16.2, 31.3, 39.8 (d, $J_{C\text{-}F}$ = 1.3 Hz), 57.5, 115.6 (d, $J_{C\text{-}F}$ = 21.0 Hz), 117.5 (d, $J_{C\text{-}F}$ = 21.5 Hz), 126.6 (d, $J_{C\text{-}F}$ = 2.7 Hz), 132.0 (d, $J_{C\text{-}F}$ = 8.6 Hz), 139.5 (d, $J_{C\text{-}F}$ = 7.5 Hz), 164.1 (d, $J_{C\text{-}F}$ = 244 Hz) ppm
^{19}F NMR:	Instrument:	Bruker Avance-400
	Field strength:	376 MHz
	Solvent:	D_2O
	Spectral data:	δ -113.5 ppm
Melting point:		130-133 °C
Microanalysis:	Found:	C = 59.0%; H = 7.5%; N = 6.9%; Cl% = 17.1%, F= 9.5% (June 2013)
	Calculated:	C = 59.0%; H = 7.4%; N = 6.9%; Cl% = 17.4%, F = 9.3% (Calculated for $C_{10}H_{15}FNCl$)