Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

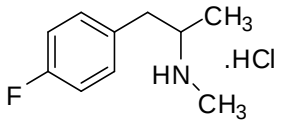
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D934: (±)-4-Fluoromethamphetamine hydrochloride

Report ID: D934.2020.04

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

Molecular Weight: 203.7 g/mol (HCl) 167.2 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
08-D-12	52063-62-4 (HCl) 351-03-1 (base)	98.7 ± 1.4%

IUPAC name: 1-(4-Fluorophenyl)-N-methyl-2-propanamine hydrochloride

Expiration of certification: The property values are valid till 30 October 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 crystalline solid 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 certified reference material is suitable for use as a primary calibrator.

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.

Metrological traceability: The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance approach all impurities are quantified as a mass fraction and subtracted from 100%. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

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.

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.
20 September 2022

This report supersedes any issued prior to 20 September 2022.

NATA Accreditation No. 198 / Corporate Site No. 14214.

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 Karl Fischer analysis, ^{1}H NMR spectroscopy and elemental microanalysis.

QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz
	Solvent:	D_2O (4.79 ppm)
	Internal standard:	Maleic acid (98.8% mass fraction)
	Initial analysis:	Mean (1.3 ppm) = 98.6%, s = 0.02% (4 sub samples, December 2020)
GC-FID:	Instrument:	Agilent 6890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	60 °C (1 min), 5 °C/min to 140 °C, 45 °C/min to 300 °C (2 min)
	Injector:	250 °C
	Detector Temp:	300 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component as the free base:	
	Initial analysis:	Mean = 99.2%, s = 0.04% (10 sub samples in duplicate, November 2008)
	Re-analysis:	Mean = 99.0%, s = 0.04% (10 sub samples in duplicate, February 2009)
GC-FID:	Instrument:	Varian CP3800
	Column:	VF-1ms, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	60 °C (1 min), 20 °C/min to 110 °C (5 min), 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 = 98.8%, s = 0.06% (5 sub samples in duplicate, February 2010)
GC-FID:	Instrument:	Varian CP3800 or Agilent 7890
	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	60 °C (3 min), 20 °C/min to 100 °C (6 min), 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 = 99.0%, s = 0.01% (5 sub samples in duplicate, February 2011)
	Re-analysis:	Mean = 98.7%, s = 0.09% (5 sub samples in duplicate, December 2013)
	Re-analysis:	Mean = 98.7%, s = 0.04% (5 sub samples in duplicate, March 2014)
	Re-analysis:	Mean = 99.0%, s = 0.2% (5 sub samples in duplicate, March 2015)
	Re-analysis:	Mean = 98.9%, s = 0.02% (5 sub samples in duplicate, January 2018)
	Re-analysis:	Mean = 99.1%, s = 0.03% (5 sub samples in duplicate, October2020)
Karl Fischer analysis:		Moisture content < 0.8% mass fraction (October 2008, January 2010, February 2010, December 2013) Moisture content 0.8% mass fraction (March 2014) Moisture content ≤ 0.5% mass fraction (March 2015, January 2018 and October 2020)
Thermogravimetric analysis:		Volatile content not determined due to the nature of the material

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	VF-1MS, 14.9 m × 0.25 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 5 °C/min to 140 °C, 45 °C/min to 300 °C (2 min)
	Injector:	250°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 (7.6 min):	109 (12), 83 (5), 58 (100), 56 (7), 42 (5) *m/z*
ESI-MS:	Instrument:	Micromass Quatro Micro
	Operation:	Positive ion mode, direct infusion at 5 μL/min
	Ionisation:	ESI spray voltage at 3.2 kV positive ion
	EM voltage:	500 V
	Cone voltage:	20 V
	Peak:	168 $(M+H)^+$ *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. MeOH/NH_3 (100/1.5) Single spot observed, R_f = 0.40. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm^{-1}, KBr powder
	Peaks:	2972, 2735, 2452, 1915, 1597, 1501, 1220, 1061, 818, 764, 548, 477 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX600
	Field strength:	600 MHz
	Solvent:	MeOH-d_4 (3.30 ppm)
	Spectral data:	δ 1.23 (3H, d, *J* = 6.5 Hz), 2.72 (3H, s), 2.76 (1H, dd, *J* = 9.5, 13.6 Hz), 3.16 (1H, dd, *J* = 5.0, 13.6 Hz), 3.41-3.49 (1H, m), 7.06 (2H, m), 7.28-7.33 (2H, m) ppm
^{13}C NMR:	Instrument:	Bruker DMX600
	Field strength:	151 MHz
	Solvent:	MeOH-d_4 (49.0 ppm)
	Spectral data:	δ 15.6, 30.9, 39.2, 57.7, 116.6 (d, J_{CF} = 20.7 Hz), 132.3 (d, J_{CF} = 8.0 Hz), 133.1 (d, J_{CF} = 2.3 Hz), 163.5 (d, J_{CF} = 244.4 Hz) ppm
^{19}F NMR:	Instrument:	Bruker DMX600
	Field strength:	565 MHz
	Solvent:	CD_3OD
	Spectral data:	δ -117.56 ppm
Melting point:		110-114 °C
Microanalysis:	Found:	C = 58.9%; H = 7.6%; N = 7.0% (October 2008)
	Calculated:	C = 59.0%; H = 7.4%; N = 6.9% (Calc for $C_{10}H_{15}ClFN$)