



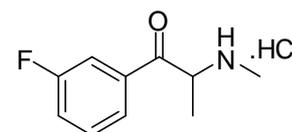
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D947b: (\pm)-3-Fluoromethcathinone hydrochloride

Report ID: D947b.2023.01

Chemical Formula: $C_{10}H_{12}FNO.HCl$

Molecular Weight: 217.7 g/mol (HCl), 181.2 g/mol (base)



Certified value

| Batch No. | CAS No. | Purity (mass fraction) |
|-----------|---------|------------------------|
| 10-D-15 | N/A | 99.4 ± 0.4% |

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

Synonyms: 1-(3-Fluorophenyl)-2-methylaminopropan-1-one hydrochloride
3-FMC.HCl

Expiration of certification: The property values are valid till 5 June 2028, 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 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 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 4 °C 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 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 measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from long term 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 HPLC with UV detection 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.
7 June 2023

This report supersedes any issued prior to 7 June 2023.

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 purity value was obtained from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (qNMR). The techniques used in the mass balance approach include GC-FID, HPLC with UV detection thermogravimetric analysis, Karl Fischer analysis and ¹H and ¹⁹F NMR spectroscopy. The purity value is calculated as per Equation 1.

$$\text{Purity} = (100\% - I_{\text{ORG}}) \times (100\% - I_{\text{VOL}} - I_{\text{NVR}}) \quad \text{Equation 1}$$

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue

The purity estimate by QNMR was obtained using a combination of the three-proton doublet at 1.45 ppm and one proton quartet at 5.2 ppm against a certified internal standard of dimethyl terephthalate.

Supporting evidence is provided by qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

| | |
|-----------------------------|---|
| HPLC: | Instrument: Shimadzu Model LC-20AB, SIL-20A HT autosampler Column: Ascentis C-18, 2.7 µm (4.6 mm x 150 mm) Mobile Phase: Acetonitrile/Milli-Q water (80:20) 0.05% TFA was present in both aqueous and organic phases. Flow rate: 0.4 mL/min Detector: Waters PDA 996 operating at 242 nm Relative peak area of the main component: Initial analysis: Mean = 99.9%, s = 0.01% (10 sub samples in duplicate, October 2010) |
| GC-FID: | Instrument: Agilent 6890 Column: HP-1, 30 m x 0.32 mm I.D. x 0.25 µm Program: 100 °C (1 min), 20 °C/min to 160 °C (10 min), 30 °C/min to 300 °C (3 min) Injector: 200 °C Detector Temp: 320 °C Carrier: Helium Split ratio: 20/1 Relative mass fraction of the main component as the <i>N</i> -acetyl derivative: Initial analysis: Mean = 99.9%, s = 0.14% (5 sub samples in duplicate, November 2010) Re-analysis: Mean = 99.5%, s = 0.04% (5 sub samples in duplicate, March 2013) Re-analysis: Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, February 2016) Re-analysis: Mean = 99.5%, s = 0.07% (5 sub samples in duplicate, January 2019) |
| GC-FID: | Instrument: Varian CP-3800 Column: HP-1 or HP-5, 30 m x 0.32 mm I.D. x 0.25 µm Program: 100 °C (1 min), 20 °C/min to 160 °C (10 min), 20 °C/min to 300 °C (3 min) or to 280 °C (5 min). Injector: 200 °C Detector Temp: 320 °C Carrier: Helium Split ratio: 20/1 Relative mass fraction of main component as the <i>N</i> -acetyl derivative: Initial analysis: Mean = 99.5%, s = 0.13% (5 sub samples in duplicate, November 2010) Re-analysis: Mean = 99.5%, s = 0.14% (5 sub samples in duplicate, September 2011) Re-analysis: Mean = 99.7%, s = 0.08% (5 sub samples in duplicate, June 2023) |
| Thermogravimetric analysis: | Non volatile residue < 0.2 % mass fraction (November 2010). The volatile content (e.g. organic solvents and/or water) could not be analysed accurately because of the inherent volatility of the material. |
| Karl Fischer analysis: | Moisture content ~ 0.4% mass fraction (February 2016 and January 2019) Moisture content ~ 0.3% mass fraction (September 2021 and May 2023) |
| QNMR: | Instrument: Bruker Avance-400 Field strength: 400 MHz Solvent: DMSO-d ₆ (2.50 ppm) Internal standard: Dimethyl terephthalate (100% m/m) Initial analysis: Mean (1.45 ppm) = 99.8%, s = 0.5% (5 sub samples, September 2010) Initial analysis: Mean (5.20 ppm) = 99.8%, s = 0.5% (5 sub samples, September 2010) |

Spectroscopic and other characterisation data

| | | |
|----------------------|--|---|
| ESI-MS: | Instrument: Operation: Ionisation: EM voltage: Cone voltage: Peak: | Micromass Quatro Micro Negative ion mode, direct infusion at 5 µL/min ESI spray voltage at 3.2 kV positive ion 650 V 14 V 182.2 (M+H ⁺) <i>m/z</i> |
| HS-GC-MS: | Instrument: Column: Program: Injector: Transfer line temp: Carrier: Split ratio: Solvents detected: | Agilent 6890/5973/G1888 DB-624, 30 m x 0.25 mm I.D. x 1.4 µm 50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min) 150 °C 280 °C Helium, 1.2 mL/min 50/1 Acetone and diethyl ether |
| IR: | Instrument: Range: Peaks: | Biorad FTS300MX FT-IR 4000-400 cm ⁻¹ , KBr powder 2950, 2739, 2690, 2442, 1700, 1585, 1474, 1436, 1261, 1097, 1017, 897, 797 cm ⁻¹ . |
| ¹ H NMR: | Instrument: Field strength: Solvent: Spectral data: | Bruker Avance-400 400 MHz D ₂ O (4.79 ppm) δ 1.63 (3H, d, <i>J</i> = 7.3 Hz), 2.84 (3H, s), 5.11 (1H, q, <i>J</i> = 7.3 Hz), 7.50 (1H, dddd, <i>J</i> = 0.9, 2.6, 8.5, 8.5 Hz), 7.62 (1H, ddd, <i>J</i> = 5.6, 8.2, 8.2 Hz), 7.73 (1H, dddd, <i>J</i> = 1.7, 1.7, 1.7, 9.3 Hz), 7.83 (1H, dddd, <i>J</i> = 1.0, 1.0, 1.0, 7.8 Hz) ppm Acetone and diethyl ether were both observed at 0.01% mass fraction in the ¹ H NMR. |
| ¹³ C NMR: | Instrument: Field strength: Solvent: Spectral data: | Bruker Avance-400 101 MHz D ₂ O δ 15.1, 31.0, 59.8, 115.3 (d, <i>J</i> _{C-F} = 23.1 Hz), 122.2 (d, <i>J</i> _{C-F} = 21.1 Hz), 125.0 (d, <i>J</i> _{C-F} = 3.0 Hz), 131.3 (d, <i>J</i> _{C-F} = 8.0 Hz), 134.2 (d, <i>J</i> _{C-F} = 7.0 Hz), 162.6 (d, <i>J</i> _{C-F} = 246.5 Hz), 196.4 (d, <i>J</i> _{C-F} = 3.0 Hz) ppm |
| ¹⁹ F NMR: | Instrument: Field strength: Solvent: Spectral data: | Bruker Avance-400 376 MHz D ₂ O δ -111.8 (1F, s) ppm A fluorinated impurity was observed at 2.6% mass fraction in the ¹⁹ F NMR. |
| Microanalysis: | Found: Calculated: | C = 55.5%; H = 6.1%; N = 6.4% (September 2010) C = 55.2%; H = 6.0%; N = 6.4% (Calculated for C ₁₀ H ₁₃ ClFNO) |