

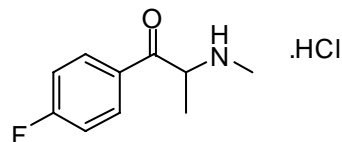


**CERTIFIED REFERENCE MATERIAL**  
**CERTIFICATE OF ANALYSIS**

**Report ID: D969.2013.01**

Compound Name: **4'-Fluoromethcathinone hydrochloride**  
Collection Number: D969  
Chemical Formula: C<sub>10</sub>H<sub>12</sub>FNO.HCl  
CAS Number: 7589-35-7  
Structure:

Description: Off white solid  
Batch Number: 11-D-09  
Molecular Weight: 217.7 (HCl), 181.2 (base)  
Release date: 5<sup>th</sup> August 2011



Synonyms: 1-(4-Fluorophenyl)-2-(methylamino)-1-propanone hydrochloride  
4'-Fluoro-2-(methylamino)-propiofenone hydrochloride  
4-FMC.HCl, Flephedrome

Purity (mass fraction): 99.5 ± 2.1% (95% coverage interval)

Purity estimate obtained from traditional analytical techniques. The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by GC-FID, thermogravimetric analysis, Karl Fischer analysis and <sup>1</sup>H NMR analysis. Supporting evidence is provided by HPLC-UV, quantitative nuclear magnetic resonance (QNMR), elemental microanalysis and headspace GC-MS analysis of occluded solvents. The purity estimate by QNMR was obtained using the three proton doublet against a certified internal standard of maleic acid.

GC-FID:	Instrument:	Varian CP3800
N-Acetyl	Column:	HP-1, 29.5 m x 0.32 mm x 0.25µm
	Program 1:	122 °C (20 min), 15 °C/min to 300 °C (3 min)
	Program 2:	150 °C (10 min), 10 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Carrier:	Helium
		Detector Temp: 320 °C
		Split ratio: 20/1
	Relative peak area response of main component:	
Program 1	Initial analysis:	Mean = 99.6%, s = 0.01% (7 sub samples in duplicate, July 2011)
Program 2	Initial analysis:	Mean = 99.5%, s = 0.01% (7 sub samples in duplicate, July, 2011)
HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Ascentis C-18, 2.7 µm (4.6 mm x 150 mm)
	Mobile Phase:	A: 20 mM Ammonium acetate/trifluoroacetic acid in MilliQ water, pH 3.0 B: Acetonitrile
		Gradient 0-10 min 12% B, 10-12 min 12%-60% B, 12-20 min 60% B, 20-21 min 12% B, 21-26 min 12% B
	Flow rate:	1 mL/min
	Detector:	Waters PDA 996 or 2998 operating at 254 nm
	Relative peak area response of main component:	
	Initial analysis:	Mean = 99.8%, s = 0.02% (7 sub samples in duplicate, June 2011)
	Re-analysis:	Mean = 99.7 %, s = 0.01 % (5 sub samples in duplicate, November 2012)
	Re-analysis:	Mean = 99.8%, s = 0.01 % (5 sub samples in duplicate, November 2013)
QNMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz
		Solvent: CD <sub>3</sub> OD (4.79 ppm)
	Internal standard:	Maleic acid (98.8 % mass fraction)
	Initial analysis:	Mean (1.5 ppm) = 99.1 %, s = 0.7% (5 sub samples, January 2012)
Thermogravimetric analysis:	The volatile content could not be determined due to the inherent volatility of the material. Non volatile residue < 0.2% mass fraction (July 2011).	
Karl Fischer analysis:	Moisture content 0.1% mass fraction (July 2011, May 2012 and October 2013)	

### Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 20 $\mu\text{L}/\text{min}$
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	15 V
	Peak:	182 ( $\text{M}+\text{H}^+$ ) $m/z$
GC-MS:	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m $\times$ 0.25 mm I.D. $\times$ 0.25 $\mu\text{m}$
<i>N</i> -Acetyl	Program:	90 $^{\circ}\text{C}$ (1 min), 10 $^{\circ}\text{C}/\text{min}$ to 180 $^{\circ}\text{C}$ (7 min), 30 $^{\circ}\text{C}/\text{min}$ to 300 $^{\circ}\text{C}$ (3 min)
Free base	Program:	90 $^{\circ}\text{C}$ (1 min), 5 $^{\circ}\text{C}/\text{min}$ to 200 $^{\circ}\text{C}$ , 20 $^{\circ}\text{C}/\text{min}$ to 300 $^{\circ}\text{C}$ (3 min)
	Injector:	200 $^{\circ}\text{C}$ Transfer line temp: 280 $^{\circ}\text{C}$
	Carrier:	Helium, 1.0 mL/min Split ratio: 20/1
The retention time of the <i>N</i> -acetyl derivative and free base are 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.		
<i>N</i> -Acetyl 10.7 min:	223 ( $\text{M}^+$ , 1), 123 (10), 101 (4), 100 (62), 95 (13), 75 (6), 59 (4), 58 (100), 56 (6), 43 (11), 42 (5) $m/z$	
Free base 9.3 min:	123 (18), 96 (3), 95 (16), 75 (9), 59 (5), 58 (100), 57 (4), 56 (21) $m/z$	
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m $\times$ 0.25 mm I.D. $\times$ 1.4 $\mu\text{m}$
	Program:	50 $^{\circ}\text{C}$ (5 min), 7 $^{\circ}\text{C}/\text{min}$ to 120 $^{\circ}\text{C}$ , 15 $^{\circ}\text{C}/\text{min}$ to 220 $^{\circ}\text{C}$ (8.3 min)
	Injector:	150 $^{\circ}\text{C}$ Transfer line temp: 280 $^{\circ}\text{C}$
	Carrier:	Helium, 1.2 mL/min Split ratio: 50/1
	Solvents detected:	Toluene, dichloromethane, ethyl acetate and methyl acetate.
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 $\text{cm}^{-1}$ , KBr powder
	Peaks:	2944, 2907, 2739, 2459, 2417, 1686, 1599, 1468, 1303, 1238, 1165, 978, 880, 850, 599 $\text{cm}^{-1}$
$^1\text{H}$ NMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz Solvent: $\text{D}_2\text{O}$ (4.79 ppm)
	Spectral data:	$\delta$ 1.60 (3H, d, $J = 7.3$ Hz), 2.80 (3H, s), 5.08 (1H, quartet, $J = 7.3$ Hz), 7.31 (2H, m), 8.07 (2H, m) ppm
Toluene, ethyl acetate and methyl acetate were observed at 0.07%, 0.01% and 0.01% mass fraction respectively in $^1\text{H}$ NMR. Dichloromethane was not detected.		
$^{13}\text{C}$ NMR:	Instrument:	Bruker Avance-400
	Field strength:	101 MHz Solvent: $\text{D}_2\text{O}$
	Spectral data:	$\delta$ 15.2, 30.9, 59.5, 116.4 (d, $J_{\text{C-F}} = 23$ Hz), 128.8 (d, $J_{\text{C-F}} = 3$ Hz), 132.0 (d, $J_{\text{C-F}} = 10$ Hz), 166.7 (d, $J_{\text{C-F}} = 256$ Hz), 196.0 ppm
$^{19}\text{F}$ NMR:	Instrument:	Bruker Avance-400
	Field strength:	565 MHz Solvent: $\text{D}_2\text{O}$
	Spectral data:	$\delta$ -102.1(m) ppm
Microanalysis:	Found: C = 55.2%; H = 6.1%; N = 6.5%; Cl = 16.4%; F = 9.0% (July, 2011) Calc: C = 55.2%; H = 6.0%; N = 6.4%; Cl = 16.3%; F = 8.7% (Calculated for $\text{C}_{10}\text{H}_{12}\text{FNO}\cdot\text{HCl}$ )	

### Expiration of certification

The property values are valid till 14<sup>th</sup> November 2016, i.e. three 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.

The long-term stability of the compound in solution has not been examined.

This material has been given a shelf life of three years from the date of re-certification.

### Homogeneity assessment

The homogeneity of the material was assessed using purity assay by GC-FID on seven randomly selected 1-2 mg sub samples of the material. The material was judged to be 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.

### Metrological Traceability

The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. The purity was derived by subtraction of the mass of impurities from the mass of the reference material. Organic purity is traceable to the SI-derived coherent unit one through chromatographic separation and response factor determination of individual components. Volatile and non-volatile residue content is directly traceable to mass through use of Karl Fischer and thermogravimetric analysis. 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).

### Recommended storage

When not in use this material should be stored at or below 25 °C in a closed container in a dry, dark area.

### Intended Use

For *in vitro* laboratory analysis only.

### Caution

Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

### Legal notice

Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R Davies  
Team Leader,  
Chemical Reference Materials, NMI  
Dated: 20 November 2013

Characterisation data and property values specified in this report supersede all reports issued prior to 20<sup>th</sup> November 2013.



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