Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

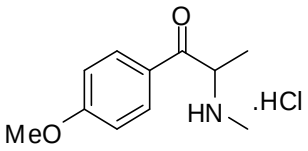
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D952: 4'-Methoxymethcathinone hydrochloride

Report ID: D952.2023.01

Chemical Formula: $C_{11}H_{15}ClNO_2$.HCl

Molecular Weight: 229.7 g/mol (HCl)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
10-D-03	**879665-92-6**	**98.9 ± 1.1%**

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

IUPAC name: 1-(4-Methoxyphenyl)-2-(methylamino)-1-propanone hydrochloride

Expiration of certification: The property values are valid till 19 October 2033, i.e. ten 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 solid prepared by 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 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 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.
10 November 2023

This report supersedes any issued prior to 10 November 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 identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, Karl Fischer analysis and ^{1}H NMR spectroscopy. 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 quantitative nuclear magnetic resonance (qNMR), HPLC with UV detection, qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800
	Column:	DB-17, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	180 °C (1 min), 2 °C/min to 210 °C (3 min), 30 °C/min to 280 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component as the *N*-acetyl derivative:	
	Initial analysis:	Mean = 99.1%, s = 0.08% (7 sub samples in duplicate, October 2023)

GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm or VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (0.5 min), 20 °C/min to 160 °C (15 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component as the *N*-acetyl derivative:	
	Initial analysis:	Mean = 99.4%, s = 0.04% (5 sub samples in duplicate, May 2011)
	Re-analysis:	Mean = 99.3%, s = 0.04% (5 sub samples in duplicate, May 2012)
	Re-analysis:	Mean = 99.2%, s = 0.06% (7 sub samples in duplicate, May 2013)
	Re-analysis:	Mean = 99.1%, s = 0.15% (5 sub samples in duplicate, March 2016)
	Re-analysis:	Mean = 99.1%, s = 0.06% (5 sub samples in duplicate, December 2019)

GC-FID:	Instrument:	Varian CP-3800
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 170 °C, 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 free base:	
	Initial analysis:	Mean = 99.6%, s = 0.02% (10 sub samples in duplicate, May 2010)

GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 170 °C, 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 componentas the free base:	
	Initial analysis:	Mean = 99.7%, s = 0.01% (10 sub samples in duplicate, May 2010)

GC-FID:	Instrument:	Varian CP-3800
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 170 °C (12 min), 10 °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 *N*-acetyl derivative:	
	Initial analysis:	Mean = 99.6%, s = 0.05% (7 sub samples in duplicate, June 2010)

HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	Ascentis C-18, 2.7 μm (4.6 mm × 150 mm)
	Column oven:	40 °C
	Mobile Phase:	Acetonitrile/MilliQ water (20:80 v/v) Both aqueous and organic phases were buffered with TFA (0.05%)
	Flow rate:	0.3 mL/min
	Detector:	Shimadzu SPD-M20A PDA operating at Max plot
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.2%, s = 0.004% (10 sub samples in duplicate, May 2010)
Karl Fischer analysis:		Moisture content < 0.2% mass fraction (April 2010) Moisture content 0.3% mass fraction (May 2011) Moisture content < 0.2% mass fraction (May 2012) Moisture content 0.3% mass fraction (March 2013) Moisture content 0.2% mass fraction (March 2016) Moisture content < 0.1% mass fraction (December 2019) Moisture content 0.1% mass fraction (October 2023)
Thermogravimetric analysis:		Non-volatile residue < 0.2% mass fraction (December 2019)
QNMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	D_2O (4.79 ppm)
	Internal standard:	Potassium hydrogen maleate (98.8% mass fraction)
	Purity estimate:	Mean = 99.3%, s = 0.5% (5 samples in duplicate, May 2010)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890/5973
	Column:	HP-1MS, 30 m × 0.25 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 170 °C, (1 min), 30 °C/min to 300°C (3 min)
	Injector:	200 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium
	Scan range:	50-550 *m/z*
	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.	
	Parent (12.1 min):	135 (16), 107 (3), 92 (9), 77 (10), 58 (100), 56 (28) *m/z*
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m × 0.25 mm I.D. × 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:	Diethyl ether
TLC:	Conditions:	Kieselgel 60F_{254}. Methanol/NH_3 (200:3) Single spot observed, R_f = 0.5. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	2924, 2716, 2464, 1677 1602, 1459, 1252, 1173, 979, 847, 764, 691, 603, 482 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Gyro-400
	Field strength:	400 MHz
	Solvent:	D_2O (4.70 ppm)
	Spectral data:	δ 1.52 (3H, d, *J* = 7.2 Hz), 2.71 (3H, s) 3.84 (3H, s), 4.99 (1H, q, *J* = 7.2 Hz), 7.04 (2H, d, *J* = 9.1 Hz), 7.93 (2H, d, *J* = 8.9 Hz) ppm
^{13}C NMR:	Instrument:	Bruker Gyro-250
	Field strength:	100 MHz
	Solvent:	D_2O
	Spectral data:	δ 15.6, 30.9, 55.7, 59.3, 114.6, 125.2, 131.6, 164.8, 195.8 ppm
Melting point:		211-215 °C
Microanalysis:	Found:	C = 57.7%; H = 7.1%; N = 6.1%; Cl = 15.6% (May, 2010)
	Calculated:	C = 57.5%; H = 7.0%; N = 6.1%; Cl = 15.4% (Calculated for $C_{11}H_{15}NO_2$.HCl)