Australian Government
Department of Industry, Science and Resources

National Measurement Institute

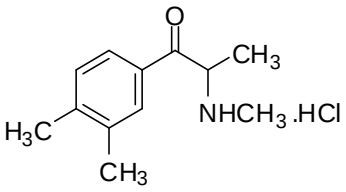
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D962: 3,4-Dimethylmethcathinone hydrochloride

Report ID: D962.2017.04

Chemical Formula: $C_{12}H_{17}NO.HCl$

Molecular Weight: 227.7 g/mol (HCl), 191.3 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
10-D-17	**1081772-06-6 (HCl)** **1082110-00-6 (base)**	**96.3 ± 5.4%**

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

IUPAC name: 1-(3,4-Dimethylphenyl)-2-(methylamino)-1-propanone hydrochloride (1:1).

Expiration of certification: The property values are valid till 8th February 2020, 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.

Description: Off-white powder 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 three 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 and 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.
20 September 2022

This report supersedes any issued prior to 20 September 2022.

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 from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (qNMR). The techniques used in the mass balance approach include GC-FID, thermogravimetric analysis, 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

The purity value by qNMR was obtained using the one-proton quartet at 5.1 ppm measured against a certified internal standard of maleic acid.

Supporting evidence is provided by HPLC with UV detection, qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument:	Varian 3800
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	165 °C (10 min), 30 °C/min to 300 °C (3 min) [2011] or 60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 280 °C (10 min) [2017]
	Injector:	250 °C or 180 °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 = 96.2%, s = 0.76% (5 sub samples in duplicate, November 2011)
	Re-analysis:	Mean = 98.7%, s = 0.14% (5 sub samples in duplicate, February 2017)
GC-FID:	Instrument:	Varian 3800
	Column:	TG-17MS, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	200 °C (10 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 = 98.7%, s = 0.04% (10 sub samples in duplicate, December 2010)
GC-FID:	Instrument:	Varian 3800
	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	180 °C (10 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 = 98.2%, s = 0.14% (10 sub samples in duplicate, December 2010)
GC-FID:	Instrument:	Varian 3800
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	165 °C (10 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 = 98.32%, s = 0.11% (10 sub samples in duplicate, December 2010)
	Re-analysis:	Mean = 96.8%, s = 0.3% (5 sub samples in duplicate, December 2012)
	Re-analysis:	Mean = 97.7%, s = 0.34% (5 sub samples in duplicate, November 2013)
HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Alltech Alltima C-18, 5 µm (4.6 mm x 150 mm)
	Mobile Phase:	Acetonitrile/1% Formic acid in MilliQ water
	Flow rate:	0.8 mL/min gradient
	Detector:	Waters PDA 996 operating at 267nm
	Relative mass fraction of the main component:	

	Initial analysis:	Mean = 96.90%, s = 0.09% (5 sub samples in duplicate, December 2011)
HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Phenomenex C-18, 5 μm (2.0 mm x 150 mm)
	Mobile Phase:	Acetonitrile/MilliQ water (17.5:82.5) 0.05% TFA was present in both aqueous and organic phases
	Flow rate:	0.40 mL/min
	Detector:	Waters PDA 996 operating at Max plot
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 97.65%, s = 0.07% (10 sub samples in duplicate, January 2011)
Karl Fischer analysis:		Moisture content 0.6% mass fraction (January 2011) Moisture content 0.9% mass fraction (November 2011) Moisture content 0.7% mass fraction (November 2012) Moisture content 0.9% mass fraction (October 2013) Moisture content 0.4% mass fraction (August 2016)
Thermogravimetric analysis:		Non volatile residue < 0.2 % mass fraction (January 2011). The volatile content (e.g. organic solvents and/or water) could not be analysed accurately because of the inherent volatility of the material.
qNMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Internal standard:	Maleic acid (98.7% mass fraction)
	Initial analysis:	Mean (5.1 ppm) = 95.08%, s = 0.88% (5 sub samples, January 2011)

Spectroscopic and other characterisation data

GC-MS:	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:	30/1
	Transfer line temp:	320 °C
	Carrier:	Helium
	Scan range:	50-550 *m/z*
	The retention time of the parent compound 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 (13.2 min):	174 (1), 133 (9), 105 (5), 77 (6), 58 (100) *m/z*
	N-Acetyl (16.5 min):	233 (M^+, 2), 133 (13), 100 (71), 58 (100) *m/z*
ESI-MS:	Instrument:	Micromass Quatro Micro
	Operation:	Positive ion mode, direct infusion at 5 µL/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	17 V
	Peak:	192 ($M+H^+$) *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
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Split ratio:	50/1
	Solvents detected:	Ethanol
TLC:	Conditions:	Kieselgel 60F_{254}. Methanol/NH_3 (100:1) Single spot observed, R_f = 0.5 Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-500 cm^{-1}, KBr powder
	Peaks:	2905, 2807, 2698, 2453, 2362, 1689, 1677, 1605, 1466, 1458, 1302, 1251, 1179, 1102, 1040, 1006, 900, 838, 829, 764, 738 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	D_2O (4.79 ppm)
	Spectral data:	δ 1.60 (3H, d, *J* = 7.3 Hz), 2.33 (3H, s), 2.35 (3H, s), 2.81 (3H, s), 5.07 (1H, q, *J* = 7.3 Hz), 7.39 (1H, d, *J* = 7.9 Hz), 7.66 (1H, dd, *J* = 1.9, 7.9 Hz), 7.79 (1H, s) ppm Ethanol estimated at 0.3% mass fraction in the ^{1}H NMR spectrum.
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	125 MHz
	Solvent:	D_2O
	Spectral data:	δ 15.5, 18.8, 19.4, 30.9, 59.5, 126.6, 129.6, 130.1, 130.3, 138.3, 146.2, 197.3 ppm
Melting point:		210-214 °C
Microanalysis:	Found:	C = 62.1%; H = 7.9%; N = 6.0%; Cl = 15.2% (January, 2011)
	Calculated:	C = 63.3%; H = 8.0%; N = 6.2%; Cl = 15.6% (Calculated for $C_{12}H_{17}NO.HCl$)