



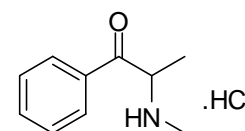
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D724b: (\pm)-Methcathinone hydrochloride

Report ID: D724b.2022.02

Chemical Formula: C₁₀H₁₃NO.HCl

Molecular Weight: 199.7 g/mol (HCl), 163.2 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
10-D-05	49656-78-2 (HCl) 5650-44-2 (base)	97.6 ± 2.1%

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

IUPAC name: 1-Phenyl-2-methylamino-1-propanone hydrochloride

Expiration of certification: The property values are valid till 21 April 2025, 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: Tan 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: 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: In the absence of long term stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated from stability trials conducted on similar materials by NMI Australia over the last ten 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.

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.
14 September 2022

This report supersedes any issued prior to 14 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 by quantitative nuclear magnetic resonance (qNMR). A combination of the three-proton singlet at 2.88 ppm and the one-proton multiplet at 5.11 ppm were measured against a certified internal standard of potassium hydrogen maleate.

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

GC-FID: Parent compound
 Instrument: HP 5890
 Column: ZB-1, 30 m × 0.32 mm I.D. × 0.25 µm
 Program: 90 °C (1 min), 10 °C/min to 110 °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 peak area of the main component:
 Initial analysis: Mean = 99.3%, s = 0.04% (10 sub samples in duplicate, September 2010)

GC-FID: *N*-Acetyl derivative
 Instrument: Varian 3800
 Column: HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
 Program: 90 °C (1 min), 20 °C/min to 150 °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 peak area of the main component:
 Initial analysis: Mean = 98.8%, s = 0.1% (10 sub samples in duplicate, September 2010)

Karl Fischer analysis: Moisture content 0.2% mass fraction (September 2010)
 Moisture content 0.9% mass fraction (June 2013)
 Moisture content 0.7% mass fraction (June 2016)
 Moisture content 0.9% mass fraction (May 2019)

Thermogravimetric analysis: Non volatile residue < 0.2% mass fraction (September 2010). 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-500
 Field strength: 500 MHz
 Solvent: D₂O (4.8 ppm)
 Internal standard: Potassium hydrogen maleate (98.6% m/m: 2010, new batch 100 % m/m: 2013)
 Initial analysis: Mean = 98.3%, s = 0.3% (5 sub samples, August 2010)
 Re-analysis: Mean = 98.4%, s = 0.03% (4 sub samples in duplicate, August 2013)
 Re-analysis: Mean = 98.5%, s = 0.3% (5 sub samples in duplicate, July 2016)

QNMR: Instrument: Bruker Avance-III-500 or Avance 400 NEO
 Field strength: 500 MHz or 400 MHz
 Solvent: Acetic acid-*d*₄ (2.03 ppm)
 Internal standard: Potassium hydrogen maleate (99.7% mass fraction)
 Initial analysis: Mean (5.11 ppm) = 97.6%, s = 0.4% (5 sub samples, September 2019)
 Initial analysis: Mean (2.88 ppm) = 97.5%, s = 0.4% (5 sub samples, September 2019)
 Re-analysis: Mean (5.11 ppm) = 97.9%, s = 0.4% (5 sub samples, April 2022)
 Re-analysis: Mean (2.88 ppm) = 97.7%, s = 0.4% (5 sub samples, April 2022)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	90 °C (1 min), 10 °C/min to 250 °C (1 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium
	Scan range:	50-550 <i>m/z</i>
	<i>N</i> -Acetyl derivative:	
	Instrument:	HP 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	110 °C (1 min), 10 °C/min to 300 °C (1 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	320 °C
	Carrier:	Helium
	Scan range:	50-550 <i>m/z</i>
	The retention times of the parent compound and <i>N</i> -acetyl derivative 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 (7.2 min):	132 (1), 105 (10), 77 (15), 58 (100), 56 (21) <i>m/z</i>
	<i>N</i> -Acetyl (9.0 min):	205 (<i>M</i> ⁺ , 1), 105 (9), 100 (72), 77 (15), 58 (100), 51 (7) <i>m/z</i>
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive/Negative ion mode, direct infusion at 10 µL/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	10 V
	Peak:	164.2 (<i>M</i> + <i>H</i> ⁺) <i>m/z</i>
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:	Isopropanol, diethyl ether
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . methanol/25% aqueous ammonia (200:3) Single spot observed, <i>R</i> _f = 0.59
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000 - 400 cm ⁻¹ , KBr powder
	Peaks:	2908, 2797, 2713, 2454, 1692, 1597, 1593, 1468, 1360, 1301, 1246, 976, 900, 700, 438 cm ⁻¹
¹ H NMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz
	Solvent:	MeOH- <i>d</i> ₄ (3.31 ppm)
	Spectral data:	δ 1.59 (3H, d, <i>J</i> = 7.3 Hz), 2.80 (3H, s), 5.17 (1H, q, <i>J</i> = 7.2 Hz), 7.60 (1H, m), 7.73 (1H, m), 8.07 (1H, m) ppm
¹³ C NMR:	Instrument:	Bruker Avance-400
	Field strength:	101 MHz
	Solvent:	MeOH- <i>d</i> ₄ (49 ppm)
	Spectral data:	δ 16.2, 31.8, 60.6, 130.0, 130.4, 134.2, 136.0, 197.1 ppm
Melting point:		173-179 °C
Microanalysis:	Found:	C = 60.3%; H = 7.2%; N = 7.1% (August, 2010)
	Calculated:	C = 60.2%; H = 7.0%; N = 7.0% (Calculated for C ₁₀ H ₁₃ NO.HCl)