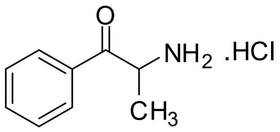
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D929b: Cathinone hydrochloride



Report ID: D929b.2021.03 (Bottled 150827)

Chemical Formula: $C_9H_{11}NO.HCl$

Molecular Weight: 185.7 g/mol (HCl), 149.2 g/mol (base)

Certified value

Batch No.	CAS No.	Purity (mass fraction)
14-D-15	**16735-19-6 (HCl)** **5265-18-9 (base)**	**99.6 ± 1.5%**

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

IUPAC name: 2-Amino-1-pheny1-1-propanone hydrochloride

Expiration of certification: The property values are valid till 19 May 2026, i.e. 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, 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 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 HPLC with UV and/or ELS 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.

Caution: 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.
25 November 2022

This report supersedes any issued prior to 19 September 2022.

NATA Accreditation No. 198 / Corporate Site No. 14214.

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 HPLC with UV detection 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 estimate by qNMR was obtained using a combination of the three proton doublet at 1.55 ppm against a certified internal standard of maleic acid

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

HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	Ascentis C-18, 2.7 µm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	Methanol/MilliQ water (40:60) 0.05% TFA was present in aqueous phase
	Flow rate:	0.25 mL/min
	Detector:	Shimadzu SPD-M20A PDA operating at 245 nm
	Relative peak area of main component:	
	Initial analysis:	Mean = 99.8%, s = 0.01% (10 sub samples in duplicate, July 2014)
	Re-analysis:	Mean = 100.0%, s = 0.01% (5 sub samples in duplicate, August 2015)
	Re-analysis:	Mean = 100.0%, s = 0.01% (5 sub samples in duplicate, August 2018)
	Re-analysis:	Mean = 100.0%, s = 0.01% (5 sub samples in duplicate, May 2021)
Karl Fischer analysis:		Moisture content 0.3% mass fraction (July 2014 and 2015) Moisture content 0.15% mass fraction (June 2018) Moisture content 0.16% mass fraction (May 2021)
QNMR:	Instrument:	Bruker Avance-III
	Field strength:	500 MHz
	Solvent:	D_2O (4.79 ppm)
	Internal standard:	Maleic acid (98.7% mass fraction)
	Initial analysis:	Mean (1.55ppm) = 99.6%, s = 0.7% (5 sub samples, July 2014)

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: 100 °C (1 min), 10 °C/min to 200 °C, 20 °C/min to 300 °C (5 min)
Injector: 250 °C
Transfer line temp: 280 °C
Carrier: Helium, 1.0 mL/min
Split ratio: 20/1

The retention time of the free base derivative is 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.

Free base (5.8 min): 134 (4), 106 (12), 104 (32), 78 (11), 77 (49), 51 (28), 44(100) *m/z*

ESI-MS:
Instrument Micromass Quatro Micro
Operation: Positive ion mode, direct infusion at 5 µL/min
Ionisation: ESI spray voltage at 3.2 kV positive ion
EM voltage: 500 V
Cone voltage 15 V
Peak: 150 $(M+H^+)$ *m/z*

TLC:
Conditions: Kieselgel 60F_{254}. Ethyl acetate/diethyl amine (100/1)
Single spot observed, $R_f = 0.13$. Visualisation with UV at 254 nm

IR:
Instrument: Biorad FTS3000MX FT-IR
Range: 4000-400 cm^{-1}, KBr powder
Peaks: 2981, 2937, 2891, 2792, 2709, 2608, 2590, 2513, 1998, 1690, 1598, 1501, 1456, 1245, 1219, 1002, 976, 791, 700 cm^{-1}

^{1}H NMR:
Instrument: Bruker Avance III 500
Field strength: 500 MHz
Solvent: D_2O (4.79 ppm)
Spectral data: δ 1.57 (3H, d, J = 7.4 Hz), 5.18 (1H, q, J = 7.3 Hz), 7.59 (2H, m), 7.74 (1H, m), 7.99 (2H, m) ppm

^{13}C NMR:
Instrument: Bruker Avance III 500
Field strength: 126 MHz
Solvent: D_2O
Spectral data: δ 16.6, 51.9, 128.8, 129.2, 132.2, 135.2, 198.0 ppm

Microanalysis:
Found: C = 58.2%; H = 6.5%; N = 7.5%; Cl = 19.1% (July, 2014)
Calculated: C = 58.2%; H = 6.5%; N = 7.5%; Cl = 19.1% (Calculated for $C_9H_{11}NO.HCl$)