



CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

Report ID: D929b.2018.01 (Bottled 150827)

This batch of bottles was prepared from the bulk material on 27th August 2015.

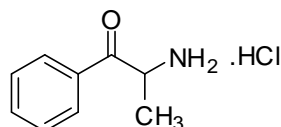
Compound Name: **Cathinone hydrochloride**

Collection Number: D929b

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

CAS Number: 16735-19-6

Structure:



Description: Off white solid

Batch Number: 14-D-15

Molecular Weight: 185.7 (HCl), 149.2 (base)

Release date: 12th August 2014

Synonyms: *dl*-Cathinone hydrochloride
(±)- α -Aminopropiophenone hydrochloride
2-Amino-1-phenyl-1-propanone hydrochloride

Purity (mass fraction): $99.6 \pm 2.5\%$ (95% coverage interval)

The purity value was obtained from quantitative nuclear magnetic resonance (qNMR). 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 HPLC-UV, Karl Fischer analysis and elemental microanalysis.

HPLC: Instrument: Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
Column: Ascentis C-18, 2.7 μ m (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 response 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)

Karl Fischer analysis: Moisture content 0.3% mass fraction (July 2014 and 2015)
Moisture content 0.15% mass fraction (June 2018)

QNMR: Instrument: Bruker Avance-III
Field strength: 500 MHz Solvent: D₂O (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 μ m
	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 μ L/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 ₂ O (4.79 ppm)
	Spectral data:	δ 1.57 (3H, d, <i>J</i> = 7.4 Hz), 5.18 (1H, q, <i>J</i> = 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 ₂ O
	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)	
	Calc: C = 58.2%; H = 6.5%; N = 7.5%; Cl = 19.1% (Calculated for C ₉ H ₁₁ NO.HCl)	

Expiration of certification

The property values are valid till 10th August 2021, 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 demonstrated stability over a minimum period of three years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from annual stability trials.

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.

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 4 °C in a closed container in a dry, dark area.

Intended use

This certified reference material may be used for instrument calibration.

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: 14 August, 2018.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 14th August 2018.