



CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

Report ID: D977.2018.01

Compound Name: **3,4-Methylenedioxy-*N,N*-dimethylcathinone hydrochloride**

Description: Beige solid

Collection Number: D977

Chemical Formula: C₁₂H₁₅NO₃.HCl

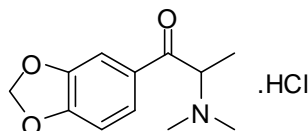
CAS Number: 109367-07-9

Structure:

Batch Number: 11-D-28

Molecular Weight: 257.7

Release date: 15th February 2012



Synonyms: 1-(1,3-Benzodioxol-5-yl)-2-(dimethylamino)-1-propanone hydrochloride

Purity (mass fraction): 98.5 ± 2.0% (95% coverage interval)

The purity value was obtained from a combination of traditional analytical techniques. The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by GC-FID thermogravimetric analysis, Karl Fischer analysis and ¹H NMR. Supporting evidence is provided by headspace GC-MS analysis of occluded solvent and elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890N
	Column:	HP-1, 30 m x 0.32 mm I.D. x 0.25 µm
	Program:	100 °C (1 min), 10 °C/min to 200 °C, 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Carrier:	Helium
		Detector Temp: 320 °C
		Split ratio: 20/1
	Relative peak area response of main component as the free base:	
	Initial analysis:	Mean = 99.0%, s = 0.11% (6 sub samples in duplicate, February 2013)
	Re-analysis:	Mean = 99.0%, s = 0.03% (5 sub samples in duplicate, January 2014)
HPLC:	Re-analysis:	Mean = 98.7%, s = 0.10% (5 sub samples in duplicate, November 2014)
	Re-analysis:	Mean = 98.8%, s = 0.10% (4 sub samples in duplicate, October 2015)
	Re-analysis:	Mean = 99.0%, s = 0.06% (5 sub samples in duplicate, August 2018)
	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20A HT autosampler
	Column:	Alltech Alltima C-18, 5 µm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	MilliQ water/methanol (50:50)
		The aqueous phase was buffered at pH 2.2 using formic acid
	Flow rate:	0.2 mL/min
	Detector:	Shimadzu SPD-M20A Photodiode Array Detector operating at 321 nm
Thermogravimetric analysis:	Relative peak area response of main component:	
	Initial analysis:	Mean = 99.9%, s = 0.04% (9 sub samples in duplicate, December 2011)
	Re-analysis:	Mean = 99.98%, s = 0.01% (5 sub samples in duplicate, December 2012)
The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material.		
Non volatile residue < 0.2% mass fraction (December 2011). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material.		
Karl Fischer analysis:	Moisture content < 0.3% mass fraction (December 2011 – October 2015, August 2018)	

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:	90 °C (1 min), 10 °C/min to 180 °C (7 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Carrier:	Helium, 1.0 mL/min
		Transfer line temp: 280 °C
		Split ratio: 20/1
	The retention time of the free base 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 (12.5 min): 149 (4), 121 (2), 91 (2), 72 (100), 70 (5), 65 (4), 56 (3), 42 (4) <i>m/z</i>	
ESI -MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 µL/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	25 V
	Peak:	222.3 (M+H ⁺) <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
	Carrier:	Helium, 1.2 mL/min
		Transfer line temp: 280 °C
		Split ratio: 50/1
	Solvents detected:	Dichloromethane
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Chloroform/methanol/diethyl amine (19:1:1) Single spot observed, R _f = 0.67. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm ⁻¹ , KBr powder
	Key peaks:	3331, 2969, 2897, 2793, 2631, 2057, 1883, 1674, 1606, 1460, 1386, 1262, 1126, 1025, 934, 868, 748 cm ⁻¹
¹ H NMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz
	Spectral data:	Solvent: D ₂ O (4.79 ppm) δ 1.63 (3H, d, <i>J</i> = 7.2 Hz), 3.00 (6H, s), 5.13 (1H, q, <i>J</i> = 7.2 Hz), 6.09 (2H, d, <i>J</i> = 3.6 Hz), 6.96 (1H, d, <i>J</i> = 8.2 Hz), 7.37 (1H, d, <i>J</i> = 1.6 Hz), 7.64 (1H, dd, <i>J</i> = 1.6, 8.2 Hz) ppm Dimethylamine hydrochloride estimated at 0.07% (2011), 0.09% (2012) and 3,4-methylenedioxy- <i>N</i> -methylcathinone hydrochloride estimated at 1.5% (2011), 1.0% (2012) mass fractions were observed in the ¹ H NMR. Dichloromethane was not observed.
¹³ C NMR:	Instrument:	Bruker DMX-600
	Field strength:	150 MHz
	Spectral data:	Solvent: D ₂ O δ 14.8, 40.7, 43.0, 65.9, 102.7, 107.8, 108.6, 126.6, 126.9, 148.3, 153.6, 195.0 ppm
Microanalysis:	Found: C = 56.1%; H = 6.4%; N = 5.4% (December, 2011) Calc: C = 55.9%; H = 6.3%; N = 5.4% (Calculated for C ₁₂ H ₁₅ NO ₃ .HCl)	

Expiration of certification

The property values are valid till 27th 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.

In the absence of 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 10 years.

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 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.

Recommended storage

When not in use this material should be stored at or below 25 °C in a closed container protected from ambient moisture and light.

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: 10 September 2018

Characterisation data and property values specified in this report supersede those in all reports issued prior to 10th September 2018.