Australian Government
Department of Industry, Science and Resources

National Measurement Institute

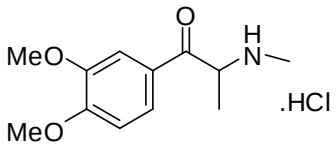
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D1062: (±)-3,4-Dimethoxymethcathinone hydrochloride

Report ID: D1062.2015.05

Chemical Formula: $C_{12}H_{17}NO_3$.HCl

Molecular Weight: 259.7 g/mol (HCl), 223.3 g/mol (base)



Property value

Batch No.	CAS No.	Purity estimate
15-D-18	22930-82-1 (HCl) 802301-79-7 (base)	95.6 ± 1.9%

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-Dimethoxyphenyl)-2-(methylamino)-1-propanone hydrochloride (1:1).

Expiration of certification: The property values are valid till 3 July 2018, i.e. three years from the date of 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 material will be re-tested on an annual basis to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

Description: White 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 reference material is recommended for qualitative analysis only.

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.

Stability: In the absence of long term stability data the stability of this material has been judged 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 eight 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.
12 October 2022

This report supersedes any issued prior to 28 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. Impurities of related structure were assessed by GC-FID. The purity estimate was obtained by mass balance from a combination of traditional analytical techniques, including 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.

Supporting evidence is provided by elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	120 °C (1 min), 10 °C/min to 240 °C, 20 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area response of main component as the *N*-acetyl derivative:	
	Initial analysis:	Mean = 99.8%, s = 0.03% (8 sub samples in duplicate, July 2015)
Karl Fischer analysis:		Moisture content 2.9% mass fraction (July 2015)
Thermogravimetric analysis:		Volatile content 2.0% and non-volatile residue 0.2% mass fraction (July 2015)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	HP-1MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 300 ºC (3 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention times of the free base and *N*-acetyl derivative are reported along 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 (15.5 min):	223 (M^+, 1), 165 (22), 137 (5), 122 (5), 107 (7), 94 (4), 79 (18), 58 (100) *m/z*
	N-Acetyl (18.4 min):	265 (M^+, 11), 165 (32), 137 (3), 122 (2), 100 (68), 79 (7), 58 (100) *m/z*
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:	10 V
	Peak:	224.5 $[M+H]^+$ *m/z*
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	2971, 2702, 1670, 1585, 1514, 1267, 1243, 1156, 1092, 1023, 1012, 866, 828, 767 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 1.45 (3H, d, *J* = 7.1 Hz), 2.57 (3H, s), 3.84 (3H, s), 3.88 (3H, s), 5.17 (1H, q, *J* = 7.1 Hz), 7.15 (1H, d, *J* = 8.5 Hz), 7.50 (1H, d, *J* = 2.0 Hz), 7.73 (1H, dd, *J* = 2.0, 8.5 Hz), 9.36 (2H, br s) ppm
	Tetrahydrofuran (1.0%), ethanol (0.1%) and acetone (0.01%) estimated mass fraction was observed in the ^{1}H NMR	
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	DMSO-d_6 (39.52 ppm)
	Spectral data:	δ 16.0, 30.7, 55.7, 56.0, 57.9, 110.7, 111.3, 124.0, 125.7, 149.0, 154.3, 194.7 ppm
Melting point:		213-215 °C
Microanalysis:	Found:	C = 54.2%; H = 7.1%; N = 5.3%; Cl = 13.2% (July 2015)
	Calculated:	C = 53.9%; H = 7.1%; N = 5.2%; Cl = 13.3% (Calculated for $C_{12}H_{17}NO_3$.HCl including 2.9% water)