



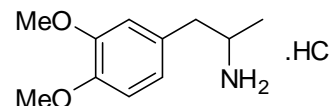
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D453b: (\pm)-3,4-Dimethoxyamphetamine hydrochloride

Report ID: D453b.2022.01

Chemical Formula: C₁₁H₁₇NO₂.HCl

Molecular Weight: 231.8 g/mol (HCl), 195.3 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
11-D-21	13078-75-6 (HCl) 120-26-3 (base)	92.0 \pm 1.4%

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-propanamine hydrochloride.

Expiration of certification: The property values are valid till 14 January 2027, 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, and certified for identity and purity by NMIA. Packaged in amber glass bottles with a septum and crimped aluminium 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 25 °C 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%.

Stability: At the recommended storage conditions this material has demonstrated stability for a period of ten years. The measurement uncertainty includes components for long term stability at the recommended storage conditions.

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.
3 February 2022

This report supersedes any issued prior to 3 February 2022

NATA logo notice: Accredited for compliance with ISO 17034. Accreditation No. 198 / Corporate Site No. 14214. The results of the tests, calibrations and/or measurements included in this document are traceable to Australian/national standards.

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 mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis, and ¹H NMR spectroscopy. The purity value is calculated as per Equation 1.

$$\text{Purity} = (100 \% - I_{\text{ORG}}) \times (100 \% - I_{\text{VOL}} - I_{\text{NVR}}) \quad \text{Equation 1}$$

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

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

GC-FID: Instrument: Varian CP3800 or Agilent 8890
 Column: VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm or HP-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 100 °C (1 min), 10 °C/min to 170 °C, 20 °C/min to 300 °C (3 min)
 Injector: 250 °C
 Detector Temp: 320 °C
 Carrier: Helium
 Split ratio: 20/1

Relative mass fraction of the main component as the free base:

Initial analysis: Mean = 99.7%, s = 0.07% (10 sub samples in duplicate, November 2011)

Re-analysis: Mean = 99.6%, s = 0.10% (5 sub samples in duplicate, January 2022)

GC-FID: Instrument: Varian CP3800
 Column: VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 100 °C (1 min), 10 °C/min to 170 °C, 20 °C/min to 300 °C (3 min)
 Injector: 250 °C
 Detector Temp: 320 °C
 Carrier: Helium
 Split ratio: 20/1

Relative mass fraction of the main component as the free base:

Initial analysis: Mean = 99.5%, s = 0.06% (10 sub samples in duplicate, November 2011)

Karl Fischer analysis: Moisture content 7.3% mass fraction (November 2011)
 Moisture content 7.4% mass fraction (February 2022)

Thermogravimetric analysis: Volatiles content 6.7% and non-volatile residue < 0.2% mass fraction (November 2011)

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
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 <i>m/z</i>
	The retention time of free base 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.	
	Free base (10.1 min):	152 (<i>M</i> ⁺ , 60), 151 (10), 137 (7), 107 (6), 44 (100) <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:	5 V
	Peak:	196.2 (<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:	Ethyl acetate, isopropanol
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . TBME/diethyl ether/diethyl amine (9:9:1) Single spot observed, <i>R</i> _f = 0.2. Visualisation with UV at 254 nm.
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm ⁻¹ , neat
	Peaks:	1519, 1264, 1147, 1025 cm ⁻¹
¹ H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	MeOH- <i>d</i> ₄ (3.31 ppm)
	Spectral data:	δ 1.28 (3H, d, <i>J</i> = 6.6 Hz), 2.78 (1H, dd, <i>J</i> = 7.9, 13.7 Hz), 2.92 (1H, dd, <i>J</i> = 6.5, 13.7 Hz), 3.52 (1H, m), 3.81 (3H, s), 3.84 (3H, s), 6.81 (1H, dd, <i>J</i> = 2.0, 8.2 Hz), 6.88 (1H, d, <i>J</i> = 2.0 Hz), 6.93 (1H, d, <i>J</i> = 8.2 Hz) ppm
	Ethyl acetate and isopropanol estimated at 1.0% and 0.1% mass fraction respectively were observed in the ¹ H NMR.	
¹³ C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	MeOH- <i>d</i> ₄ (49 ppm)
	Spectral data:	δ 18.4, 41.4, 50.4, 56.47, 56.48, 113.2, 114.0, 122.8, 130.1, 149.8, 150.7 ppm
Melting point:	144-147 °C	
Microanalysis:	Found:	C = 53.0%; H = 8.2%; N = 5.6% (November, 2021)
	Calculated:	C = 57.0%; H = 7.8%; N = 6.0% (Calculated for C ₁₃ H ₁₇ NO ₂ .HCl)
	Calculated:	C = 52.9%; H = 8.1%; N = 5.6% (Calculated for C ₁₃ H ₁₇ NO ₂ .HCl + 7.3% H ₂ O)