Australian Government
Department of Industry, Science and Resources
National Measurement Institute
NATA
WORLD RECOGNISED ACCREDITATION

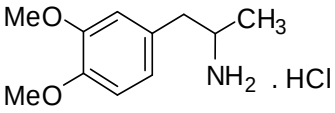
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D453: (±)-3,4-Dimethoxyamphetamine hydrochloride

Report ID: D453.2023.01

Chemical Formula: $C_{11}H_{17}NO_2$.HCl

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



Property value

Batch No.	CAS No.	Purity estimate
95-033183	13078-75-6	92.3 ± 1.3%

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 17 January 2033, i.e. ten 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: 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 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 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%.

Stability: This material has demonstrated stability over a minimum period of five 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 GC-FID on five 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.
23 January 2023

This report supersedes any issued prior to 23 January 2023.

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

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 and Karl Fischer analysis. 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 ^{1}H NMR spectroscopy and elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890
	Column:	BP-5, 25 m x 0.33 mm I.D. x 0.5 μm
	Program:	100 °C (1 min), 10 °C/min to 250 °C, 25 °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 = 99.9%, s = 0.1% (3 sub samples in duplicate, December 1995)
	Re-analysis:	Mean = 99.4%, s = 0.04% (5 sub samples in duplicate, February 2005)
	Re-analysis:	Mean = 99.4%, s = 0.13% (5 sub samples in duplicate, February 2008)
GC-FID:	Instrument:	Agilent 6890 or 8890
	Column:	HP-1, 30 m x 0.32 mm I.D. x 0.25 μm
	Program:	120 °C (0.5 min), 8 °C/min to 180 °C, 15 °C/min to 300 °C (3 min)
	Injector:	200 °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 = 0.01% (5 sub samples in duplicate, January 2011)
	Re-analysis:	Mean = 99.5%, s = 0.01% (5 sub samples in duplicate, April 2018)
	Re-analysis:	Mean = 99.6%, s = 0.01% (5 sub samples in duplicate, January 2023)
	Karl Fischer analysis:	Moisture content ca. 7.3% mass fraction (January 2008, 2011, 2012, April 2018 & January 2023)
Thermogravimetric analysis:		Volatile content 6.7% and non-volatile residue not determined (January 2008) Volatile content 7.3% and non-volatile residue < 0.1 % mass fraction (January 2011)

Spectroscopic and other characterisation data

GC-MS:	Free base:	
	Instrument:	HP5890/5970B
	Column:	HP Ultra-2, 12 m x 0.22 mm x 0.1 μm
	Program:	70 °C to 300 °C at 10 °C/min
	Injector:	230 °C,
	Split ratio:	10/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention time of the 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 (8.8 min):	195, 152, 137, 121, 107, 91, 77, 65, 44 *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Diisopropylether/diethylether/diethylamine (45/45/10) Single spot Rf = 0.2 Toluene/acetone/ethanol/conc. ammonia (40/40/6/2) Primary spot Rf = 0.3
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-500 cm^{-1}, KBr powder
	Peaks:	1519, 1264, 1147, 1025 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Gyro-600
	Field strength:	600 MHz
	Solvent:	MeOH-d_4 (3.31 ppm)
	Spectral data:	δ 1.27 (3H, d, *J* = 6.7 Hz), 2.79 (1H, dd, *J* = 7.9, 13.7 Hz), 2.93 (1H, dd, *J* = 6.6, 13.7 Hz), 3.52 (1H, m), 6.81 (1H, dd, *J* = 2.0, 8.0 Hz), 6.88 (1H, d, *J* = 2.0 Hz), 6.92 (1H, d, *J* = 8.2 Hz) ppm
^{13}C NMR:	Instrument:	Bruker Gyro-600
	Field strength:	151 MHz
	Solvent:	MeOH-d_4 (49.0 ppm)
	Spectral data:	δ 17.4, 40.4, 49.4, 54.5, 55.5, 112.3, 113.0, 121.8, 129.1, 148.8, 149.7 ppm
Melting point:		147.5 – 148.5 °C (uncorrected)
Microanalysis:	Found:	C = 52.8%, H = 8.2%; N = 5.7% (January, 2008)
	Calculated:	C = 57.0%, H = 7.8%; N = 6.0% (Calculated for $C_{11}H_{17}NO_2$.HCl)