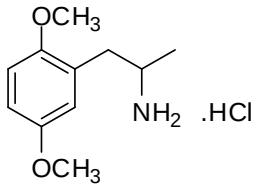
Australian Government
Department of Industry, Science and Resources

National Measurement Institute

CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D749b: (±)-2,5-Dimethoxyamphetamine hydrochloride



Report ID: D749b.2020.03

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

Molecular Weight: 231.7 g/mol (HCl), 195.3 g/mol (base)

Certified value

Batch No.	CAS No.	Purity (mass fraction)
09-D-19	24973-25-9 (HCl) 2801-68-5 (base)	99.2 ± 1.5%

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit (k = 2).

IUPAC name: 1-(2,5-Dimethoxyphenyl)-2-propanamine hydrochloride

Expiration of certification: The property values are valid till 19 June 2030, 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: 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 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%. 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).

Stability: This material has demonstrated stability over a minimum period of three 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.
15 September 2022

This report supersedes any issued prior to 15 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. The certified purity value was obtained from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (qNMR). The techniques used in the mass balance approach include 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

The certified purity value by qNMR was obtained using the one-proton doublet of doublets at 2.85 ppm measured against a certified internal standard of potassium hydrogen maleate.

Supporting evidence is provided by elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890
	Column:	HP-1, 30 m x 0.32 mm I.D. x 0.25 μm
	Program:	100 °C (0.5 min), 10 °C/min to 250°C, 30 °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:	
	Initial analysis:	Mean = 99.5%, s = 0.02% (10 sub samples in duplicate, October 2009)
	Re analysis:	Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, October 2011)
	Re analysis	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, October 2012)
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 250 °C (4 min), 20 °C/min to 300 °C (2 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.5%, s = 0.03% (10 sub samples in duplicate, October 2009)
GC-FID:	Instrument:	Varian CP-3800 or Agilent 6890
	Column:	HP-1, 30 m x 0.32 mm I.D. x 0.25 μm
	Program:	100 °C (0.5 min), 8 °C/min to 180 °C, 10 °C to 280 °C, 30 °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:	
	Initial analysis:	Mean = 99.3%, s = 0.004% (5 sub samples in duplicate, November 2010)
	Re analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, August 2015)
	Re analysis:	Mean = 99.8%, s = 0.04% (5 sub samples in duplicate, June 2020)
Karl Fischer analysis:		Moisture content ≤ 0.2% mass fraction (November 2009 and 2010, October 2011 and 2012, August 2015, June 2020)
Thermogravimetric analysis:		Volatile content not determined due to the nature of the material. Non-volatile content was found to be < 0.2% (December 2009).
QNMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	D_2O (4.79 ppm)
	Internal standard:	Potassium hydrogen maleate
	Initial analysis:	Mean (2.85 ppm) = 98.8%, s = 0.4% (5 sub samples, December 2009)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	VF-1MS, 15 m × 0.25 mm I.D. × 0.25 μm
	Program:	120 °C (10 min), 25 °C/min to 300 °C (5 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 time of the parent compound 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.	
	Parent (7.9 min):	195 (M^+, 4), 152 (100), 137 (26), 121 (11), 108 (4), 91 (10) 77 (11), 65 (10) *m/z*
ESI-MS:	Instrument:	Micromass Quatro Micro
	Operation:	Positive ion mode, direct infusion at 5 μL/min
	Ionisation:	ESI spray voltage at 3.6 kV positive ion
	EM voltage:	500 V
	Cone voltage:	20 V
	Peak:	196 ($M+H^+$) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. MeOH/NH_3 (100:1.5) Single spot observed, R_f = 0.4. Visualisation with UV at 254 nm.
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	2940, 2600, 2511, 2054, 1609, 1505, 1454, 1304, 1226, 1042, 803, 705 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	MeOH-d_4 (3.31 ppm)
	Spectral data:	δ 1.25 (3H, d, *J* = 6.6 Hz), 2.82 (1H, dd, *J* = 7.4, 13.5 Hz), 2.95 (1H, dd, *J* = 6.5, 13.3 Hz), 3.56 (1H, m), 3.75 (3H, s), 3.81 (3H, s), 6.79 (1H, d, *J* = 2.9 Hz), 6.84 (1H, dd, *J* = 3.1, 8.9 Hz), 6.93 (1H, d, *J* = 8.9 Hz) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-400
	Field strength:	100 MHz
	Solvent:	MeOH-d_4 (49.0 ppm)
	Spectral data:	δ 18.6, 36.9, 56.1, 56.2, 112.8, 114.0, 118.6, 126.2, 153.1, 155.1 ppm
Melting point:		110-114 °C
Microanalysis:	Found:	C = 57.1%; H = 7.9%; N = 6.1%; Cl = 15.3% (November 2009)
	Calculated:	C = 57.0%; H = 7.8%; N = 6.0%; Cl = 15.3% (Calculated for $C_{11}H_{17}NO_2$.HCl)