Australian Government
Department of Industry, Science and Resources

National Measurement Institute

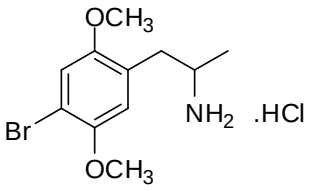
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D396b: (±)-4-Bromo-2, 5-dimethoxyamphetamine hydrochloride

Report ID: D396b.2020.03

Chemical Formula: $C_{11}H_{16}BrNO_2.HCl$

Molecular Weight: 310.6 g/mol (HCl), 274.2 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
09-D-23	**29705-96-2**	**99.1 ± 0.4%**

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

IUPAC name: (±)-1-(4-Bromo-2, 5-dimethoxyphenyl)-2-propanamine hydrochloride

Expiration of certification: The property values are valid till 1 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 solid 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 five years. The measurement uncertainty at the 95% coverage 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 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.
14 September 2022

This report supersedes any issued prior to 26 July 2022.

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

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, 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 quantitative NMR spectroscopy and elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890 or Agilent 7890
	Column:	HP-1 or HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 10 °C/min to 210 °C, 30 °C/min to 300 °C (3 min)
	Injector:	180 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.1%, s = 0.04% (10 sub samples in duplicate, October 2009)
	Re-analysis:	Mean = 99.1%, s = 0.02% (5 sub samples in duplicate, November 2011)
	Re-analysis:	Mean = 99.1%, s = 0.03% (5 sub samples in duplicate, September 2014)
	Re-analysis:	Mean = 99.0%, s = 0.06% (5 sub samples in duplicate, June 2017)
	Re-analysis:	Mean = 99.0%, s = 0.04% (5 sub samples in duplicate, June 2020)
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 = 99.2%, s = 0.04% (10 sub samples in duplicate, October 2009)
	Re-analysis:	Mean = 99.1%, s = 0.02% (5 sub samples in duplicate, November 2010)
GC-FID:	Instrument:	Varian CP3800
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	120 °C (10 min), 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:	
	Initial analysis:	Mean = 99.0%, s = 0.07% (10 sub samples in duplicate, October 2009)
QNMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	D_2O
	Internal standard:	Potassium hydrogen maleate
	Purity estimate:	Mean = 100.3%, s = 0.3% (5 samples, December 2009)
Thermogravimetric analysis:		Volatile and non-volatile content not determined due to the nature of the material
Karl Fischer analysis:		Moisture content < 0.2% mass fraction (November 2009, October 2010, October 2011 & October 2014) Moisture content < 0.1% mass fraction (July 2017, May 2020)

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Micromass Quatro Micro
	Operation:	Positive ion mode, direct infusion at 5 μL/min
	Ionisation:	ESI spray voltage at 3.60 kV positive ion
	EM voltage:	500 V
	Cone voltage:	20 V
	Peak:	276 $(M(Br^{81})+H^+)^+$ and 274 $(M(Br^{79})+H^+)^+$ *m/z*
GC-MS:	Instrument:	Agilent 6890/5973
	Column:	VF-1ms, 15 m × 0.25 mm I.D. × 0.30 μm
	Program:	120 °C (10 min), 25 °C/min to 300 °C, (5 min)
	Injector:	250 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	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.	
	12.9 min:	275 ($M^+(Br^{81})$ 2), 273 ($M^+(Br^{79})$ 2), 232 (95), 230 (100), 217 (17), 215 (18), 201 (13), 199 (12), 121 (16), 105 (24), 91 (23), 77 (48), 53 (17) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. MeOH/NH_3 (100/1.5) Single spot observed, R_f = 0.37. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	2901, 2569, 2500, 2029, 1592, 1497, 1390, 1213, 1034, 860, 792, 733 cm^1
^{1}H NMR:	Instrument:	Bruker Avance III - 400
	Field strength:	400 MHz
	Solvent:	MeOH-*d*$_4$ (3.31 ppm)
	Spectral data:	δ 1.27 (3H, d, *J* = 6.7 Hz), 2.85 (1H, dd, *J* = 7.0, 13.5 Hz), 2.95 (1H, dd, *J* = 6.8, 13.5 Hz), 3.57 (1H, m), 3.81 (3H, s), 3.84 (3H, s), 6.95 (1H, s), 7.18 (1H, s) ppm
		Isopropanol and diethyl ether quantified at 0.11% and 0.08% mass fraction by ^{1}H NMR.
^{13}C NMR:	Instrument:	Bruker Avance III – 400
	Field strength:	100 MHz
	Solvent:	MeOH-*d*$_4$ (49.0 ppm)
	Spectral data:	δ 18.6, 36.6, 49.1, 56.6, 57.4, 111.6, 116.9, 117.2, 125.6, 151.6, 153.4 ppm
Melting point:		197-200 °C
Microanalysis:	Found:	C = 42.7 %; H = 5.5 %; N = 4.5%; Cl = 11.3%; Br = 25.7% (November 2009)
	Calculated:	C = 42.5 %; H = 5.5 %; N = 4.5%, Cl = 11.4%, Br = 25.7% (Calculated for $C_{11}H_{16}BrNO_2$.HCl)