Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

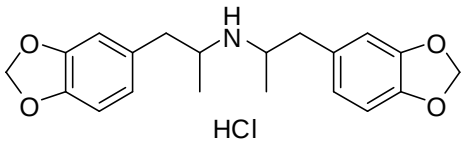
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D896: N, N-Di[1-(3,4-Methylenedioxyphenyl)-2-propyl]amine hydrochloride

Report ID: D896.2021.01

Chemical Formula: $C_{20}H_{23}NO_4.HCl$

Molecular Weight: 377.9 g/mol (HCl), 341.4 g/mol (base)



Certified value

Batch No.	CAS No.	Purity (mass fraction)
05-D-13	67668-98-8 (base)	99.3 ± 0.3%

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

IUPAC name: 1-(1,3-Benzodioxol-5-yl)-N-[1-(1,3-benzodioxol-5-yl)-2-propanyl]-2-propanamine HCl

Expiration of certification: The property values are valid till 13 August 2031 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 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%.

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 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.
23 August 2021

This report supersedes any issued prior to 23 August 2021.

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 ^{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:	Agilent 6890N or 8890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	180 °C (1 min), 30 °C/min to 250 °C (5 min), 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% (7 sub-samples in duplicate, August 2005)
	Re-analysis:	Mean = 99.5%, s = 0.01% (5 sub-samples in duplicate, November 2007)
	Re-analysis:	Mean = 99.5%, s = 0.01% (5 sub-samples in duplicate, November 2012)
	Re-analysis:	Mean = 99.6%, s = 0.01% (5 sub-samples in duplicate, August 2021)
Thermogravimetric analysis:		Volatile content < 0.1% mass fraction. Non volatile residue was not determined (August 2005, November 2006 and December 2007).
Karl Fischer analysis:		Moisture content < 0.3% mass fraction (November 2012 & August 2021)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP5890/5971A
	Column:	ZB-5, 30 m × 0.25 mm I.D. × 0.20 μm
	Program:	220 °C (2 min), 10 °C/min to 290 °C (5 min), 10 °C/min to 300 °C
	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.	
	Free base (8.9 min):	206 (98), 163 (100), 135 (48), 105 (33), 77 (27), 70 (14), 51 (11) *m/z*
TLC:	Conditions:	Kieselgel 60F254. Methanol/Conc NH_3 (200:3) Single spot observed, Rf = 0.7. Visualisation with UV at 254 nm.
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1}, KBr pellet
	Peaks:	2973, 2819, 2788, 2754, 2485, 2461, 2046, 1848, 1607, 1493, 1442, 1250, 1042, 933, 862, 806, 778 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	MeOH-*d*$_4$
	Spectral data:	δ 1.25 (6H, d, *J* = 6.6 Hz), 2.65 (2H, dd, *J* = 9.3, 13.4 Hz), 3.05 (2H, dd, *J* = 5.2, 13.6 Hz), 3.51 (2H, m), 5.94 (4H, s), 6.69 (2H, dd, *J* = 1.7, 8.0 Hz), 6.74 (2H, d, *J* = 1.6 Hz), 6.78 (2H, d, *J* = 8.0 Hz) ppm
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	125 MHz
	Solvent:	MeOH-*d*$_4$
	Spectral data:	δ 16.9, 40.2, 54.7, 102.6, 109.5, 110.3, 123.6, 130.8, 148.4, 149.6 ppm
Melting point:		261-263 °C
Microanalysis:	Found:	C = 63.3%; H = 6.3%; N = 3.7%
	Calculated:	C = 63.6%; H = 6.4%; N = 3.7% (Calculated for $C_{20}H_{23}NO_4$.HCl)