



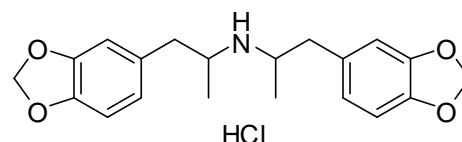
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D879: N,N-Di[1-(3,4-methylenedioxyphenyl)-2-propyl]amine hydrochloride

Report ID: D879.2021.03

Chemical Formula: C₂₀H₂₃NO₄.HCl

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



Property value

Batch No.	CAS No.	Purity estimate
05-D-16	67668-98-8 (base)	99.6 ± 0.4% (as a mixture of isomers)

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

Expiration of certification: The property values are valid till 3 February 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 reference material is recommended for qualitative analysis only.

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.

Stability: This material has demonstrated stability over a minimum period of ten years. 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- 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.
13 October 2022

This report supersedes any issued prior to 19 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. 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.

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

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

Supporting evidence is provided by elemental microanalysis.

Note: This material comprises of two diastereomers in a ratio of 24:76.

GC-FID:	Instrument:	Agilent 6890N or 7890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	180 °C (1 min), 10 °C/min to 300 °C (6 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area response of main component:	
	Initial analysis:	Mean = 99.9%, s = 0.01% (7 sub samples in duplicate, August 2005)
	Re-analysis:	Mean = 99.7%, s = 0.1% (5 sub samples in duplicate, June 2008)
	Re-analysis:	Mean = 99.8%, s = 0.1% (5 sub samples in duplicate, June 2011)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, March 2016)
	Re-analysis:	Mean = 99.9%, s = 0.04% (5 sub samples in duplicate, February 2021)
Karl Fischer analysis:	Moisture content 0.4% mass fraction (2 sub samples, June 2008)	
	Moisture content 0.3% mass fraction (2 sub samples, June 2011)	
	Moisture content 0.4% mass fraction (2 sub samples, April 2016)	
	Moisture content 0.2% mass fraction (2 sub samples, January 2021)	
Thermogravimetric analysis:	Volatile content < 0.1% (June 2007 and August 2005). Non-volatile residue was not determined due to the nature of the material.	

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
	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 the parent compound is reported along with the major peaks in the mass spectrum. The latter are reported as mass/charge ratio and (in brackets) as a percentage relative to the base peak.	
	8.9 min:	206 (100), 163 (96), 135 (46), 105 (33), 77 (24), 70 (14), 51 (9) <i>m/z</i>
	9.1 min:	206 (99), 163 (100), 135 (43), 105 (30), 77 (27), 70 (3), 51 (8) <i>m/z</i>
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Methanol/Conc NH ₃ (200:3) Single spot observed, R _f = 0.7. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR.
	Range:	4000-400 cm ⁻¹ , KBr pellet.
	Peaks:	2974, 2783, 2708, 2461, 1588, 1503, 1492, 1444, 1251, 1038, 933, 808 cm ⁻¹
¹ H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	MeOH- <i>d</i> ₄ (3.30 ppm)
	Spectral data:	δ 1.23 (3H, d, <i>J</i> = 6.5 Hz, major isomer), 1.26 (3H, d, <i>J</i> = 6.5 Hz, minor isomer) 2.65-2.72 (2H, m), 3.08-3.13 (2H, m), 3.51 (2H, m), 5.93 (4H, s), 6.69-6.78 (6H, m) ppm.
¹³ C NMR:	Instrument:	Bruker DMX-500
	Field strength:	125 MHz
	Solvent:	MeOH- <i>d</i> ₄ (49.0 ppm)
	Spectral data:	δ 13.6, 16.8, 40.1, 40.3, 54.8, 54.7, 102.5, 109.5, 109.5, 110.4, 123.6, 123.6, 130.9, 131.0, 148.4, 149.6, 149.6 ppm.
Melting point:	209-212 °C	
Microanalysis:	Found:	C = 63.5%; H = 6.4%; N = 3.7% (August 2005)
	Calculated:	C = 63.6%; H = 6.4%; N = 3.7% (Calculated for C ₂₀ H ₂₃ NO ₄ .HCl)