Australian Government
Department of Industry, Science and Resources

National Measurement Institute

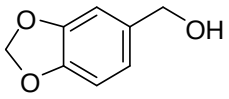
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D881: Piperonol

Report ID: D881.2021.02

Chemical Formula: $C_8H_8O_3$

Molecular Weight: 152.15 g/mol



Property value

Batch No.	CAS No.	Purity estimate
04-D-18	**495-76-1**	**98.1 ± 1.2%**

IUPAC name: 1,3-Benzodioxol-5-ylmethanol

Expiration of certification: The property values are valid till 12 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: Bright yellow solid sourced from external supplier, 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 ºC in a closed container in a dry, dark area.

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

This report supersedes any issued prior to 19 September 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. 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. 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 thermogravimetric analysis, Karl Fischer analysis and elemental microanalysis.

GC-FID**:**	Instrument:	Varian CP3800
	Column:	HP-5, 30 m x 0.32 mm I.D. x 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 250 °C (5 min), 40 °C/min to 300 °C
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 98.4 %, s = 0.07% (7 sub-samples in duplicate, October 2004)
	Re-analysis:	Mean = 98.1 %, s = 0.10% (5 sub-samples in duplicate, March 2007)
	Current re-analysis:	Mean = 98.5 %, s = 0.10% (5 sub-samples in duplicate, February 2008)
GC-FID**:**	Instrument:	Varian CP3800
	Column:	HP-5, 30 m x 0.32 mm I.D. x 0.25 μm
	Program:	110 °C (1 min), 15 °C/min to 270 °C (5 min), 30 °C/min to 280 °C (5min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 98.2 %, s = 0.09% (5 sub-samples in duplicate, February 2011)
	Re-analysis:	Mean = 98.3 %, s = 0.16% (5 sub-samples in duplicate, August 2021)
Karl Fischer analysis:		Moisture content 1.0% mass fraction (June 2005) Moisture content 0.3% mass fraction (February 2008) Moisture content 0.2% mass fraction (February 2011, August 2021)
Thermogravimetric analysis:		Non-volatile residue < 0.2% total mass fraction. Volatile content not determined due to volatility of the material (April 2005)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890 / 5973
	Column:	Zebron ZB-5, 28 m x 0.25 mm I.D. x 0.25 μm
	Program:	80 °C (1min), 15 °C/min to 100 °C, 20 °C/min to 300 °C (6 min)
	Injector:	180 °C
	Split ratio:	40/1
	Transfer line Temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention time of the material is reported along with the major peaks in the mass spectrum. The latter are reported in mass to charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (6.63 min):	152 (100), 151 (32), 135 (50), 123 (25), 122 (23), 121 (14), 93 (45), 77 (16), 65 (32), 51 (9) *m/z*
IR:	Instrument:	Biorad FTS 3000 MXFT-IR
	Range:	4000-400cm^{-1}, KBr powder
	Peaks:	3293, 2909, 1844, 1718, 1605, 1497, 1446, 1371, 1251, 1099, 1036, 921, 862, 805, 768 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 4.57 (2H, s), 5.95 (2H, s), 6.79 (1H, d, *J* = 8.0, 9.0 Hz), 6.80 (1H, bd, *J* = 9.0 Hz), 6.86 (1H, bs) ppm
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	125 MHz
	Solvent:	$CDCl_3$ (77.2 ppm)
	Spectral data:	δ 65.2, 101.0, 107.9, 108.2, 120.5, 134.8, 147.1, 147.8 ppm
Melting point:		53-55 °C
Microanalysis:	Found:	C = 63.3%, H = 5.4% (November 2005)
	Calculated:	C = 63.2%, H = 5.3% (Calculated for $C_8H_8O_3$)