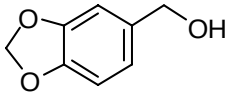
Australian Government

National Measurement Institute

REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D881.2011.02

Compound Name: **Piperonol**
Collection Number: D881
Chemical Formula: $C_8H_8O_3$
CAS Registry Number: 495-76-1
Structure:



Description: Bright yellow solid
Batch Number: 04-D-18
Molecular Weight: 152.15

Synonyms: Piperonyl alcohol (6Cl,7Cl,8Cl); 1-Hydroxymethyl-3,4-methylene-dioxybenzene; 3,4-(Methylenedioxy)benzenemethanol; 3,4-Methylenedioxybenzyl alcohol; 3,4-Methylenedioxyphenyl-methanol; 5-Hydroxymethyl-1,3-benzodioxole; Benzo[1,3]dioxol5-ylmethanol.

Purity (mass fraction): 98.1 ± 1.2 % (95 % coverage interval)

Purity estimate obtained from a combination of traditional analytical techniques. The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by GC-FID thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR. Supporting evidence is provided by 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 Temp:	250 °C	Detector Temp: 320 °C
Carrier:	Helium	Split ratio: 20/1

Relative peak area response of 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 300 °C (3 min)	
Injector Temp:	250 °C	Detector Temp: 320 °C
Carrier:	Helium	Split ratio: 20/1

Relative peak area response of main component:

Initial analysis:	Mean = 98.2 %, s = 0.09% (5 sub-samples in duplicate, February 2011)

Thermogravimetric analysis: Non-volatile residue < 0.2% total mass fraction. Volatile content not determined due to volatility of the material (April 2005)

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)

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 Temp:	180 °C Transfer line Temp: 300 °C
	Carrier:	Helium, 1.0 mL/min Split ratio: 40/1
	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 (Lit. 53 - 54 °C)
Microanalysis:		Found: C = 63.3%, H = 5.4% (November 2005)
		Calc: C = 63.2%, H = 5.3% (Calculated for $C_8H_8O_3$)

Expiration of certification
The property values are valid till 4th February 2016, i.e. five 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.

The long-term stability of the compound in solution has not been examined.

This material has demonstrated stability over a minimum period of 5 years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from annual stability trials.

Homogeneity assessment
The homogeneity of the material was assessed using purity assay by GC with flame ionisation detector on five randomly selected 1-2 mg sub samples of the material. The material was judged to be 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.

Recommended storage
When not in use, this material should be stored at or below 20 ºC in a closed container in a dry, dark area.

Intended Use
For ***in vitro*** laboratory analysis only.

Caution
Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice
Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 20 July, 2012.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 20th July 2012

105 Delhi Road North Ryde NSW 2113 PO Box 138 North Ryde NSW 1670 Tel: +61 2 9449 0111 Fax: +61 2 9449 0292 www.measurement.gov.au ABN: 74 599 608 295