

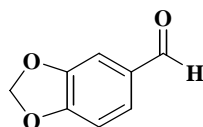


## REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D870.2014.01

Compound Name: **Piperonal**  
Collection Number: D870  
Chemical Formula:  $C_8H_6O_3$   
CAS Number: 120-57-0  
Structure:

Description: Off white solid  
Batch Number: 04-D-012  
Molecular Weight: 150.1  
Release Date: 21<sup>st</sup> December 2004



Synonyms: 1,3-Benzodioxole-5-carboxaldehyde  
3,4-(Methylenedioxy)benzaldehyde  
Piperonylaldehyde  
Dioxymethyleneprotocatechuic aldehyde

Purity (mass fraction):  $99.9 \pm 0.3\%$  (95 % coverage interval)

The purity value was 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  $^1H$  NMR analysis. Supporting evidence is provided by elemental microanalysis.

GC-FID: Instrument: Agilent 6890N  
Column: HP-1, 30 m  $\times$  0.32 mm I.D.  $\times$  0.25  $\mu$ m  
Program: 60  $^{\circ}C$  (1 min), 10  $^{\circ}C/min$  to 120  $^{\circ}C$ , 20  $^{\circ}C/min$  to 300  $^{\circ}C$   
Injector: 200  $^{\circ}C$  Detector Temp: 320  $^{\circ}C$   
Carrier: Helium Split ratio: 20/1  
Relative peak area response of main component:  
Initial analysis: Mean = 99.99%, s = 0.006% (10 sub samples in duplicate, June 2004)  
Re-analysis: Mean = 99.99%, s = 0.003% (5 sub samples in duplicate, August 2006)  
Re-analysis: Mean = 99.99%, s = 0.002% (5 sub samples in duplicate, September 2009)  
Re-analysis: Mean = 99.99%, s = 0.003% (5 sub samples in duplicate, July 2014)

Karl Fischer analysis: Moisture content < 0.1% mass fraction (August 2006 and August 2009)  
Moisture content < 0.1% mass fraction (July 2014)

### Spectroscopic and other characterization data

GC-MS:	Instrument:	HP 6890/5973
	Column:	ZB-5, 30 m × 0.25 mm I.D. × 0.20 µm
	Program:	60 °C (1 min), 10 °C/min to 250 °C (1 min)
	Injector:	220 °C
	Carrier:	Helium
		Transfer line temp: 280 °C
		Split ratio: 20/1
	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 ratios and (in brackets) as a percentage relative to the base peak.	
	11.6min: 151 (M <sup>+</sup> , 8), 150 (84), 149 (100), 121 (27), 91 (9), 63 (22), 62 (10) m/z	
TLC:	Conditions:	Kieselgel 60F <sub>254</sub> . Hexane/ethyl acetate (3/1)
		Single spot observed, R <sub>f</sub> = 0.24. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm <sup>-1</sup> , KBr pellet
	Peaks:	3330, 3000, 2920, 2852, 2795, 2044, 1863, 1679, 1599, 1488, 1449, 1256, 1096, 1037, 929, 815, 786 cm <sup>-1</sup>
<sup>1</sup> H NMR:	Instrument:	Bruker Gyro-300
	Field strength:	300 MHz
	Spectral data:	Solvent: CDCl <sub>3</sub> δ 6.06 (2H, s), 6.91 (1H, d, <i>J</i> = 7.9 Hz), 7.31 (1H, d, <i>J</i> = 1.1 Hz), 7.39 (1H, dd, <i>J</i> = 1.5, 7.9 Hz), 9.79 (1H, s) ppm
<sup>13</sup> C NMR:	Instrument:	Bruker Gyro-300
	Field strength:	75 MHz
	Spectral data:	Solvent: CDCl <sub>3</sub> δ 102.0, 106.9, 108.3, 128.6, 131.9, 148.7, 153.1, 190.2 ppm
Melting point:		36 - 37 °C (Lit 37 °C)
Microanalysis:	Found:	C = 64.1 %, H = 4.2 % (July 2004)
	Calc:	C = 64.0 %, H = 4.0 % (Calculated for C <sub>8</sub> H <sub>6</sub> O <sub>3</sub> )
Thermogravimetric analysis:		Non volatile residue < 0.2 % total mass fraction
		Volatile content not determined due to volatility of the material

### Expiration of certification

The property values are valid till 10<sup>th</sup> July 2019, 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 five 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-FID on ten 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 25 °C 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: 21 July, 2014.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 21<sup>st</sup> July 2014.