Australian Government

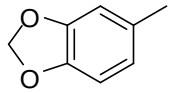
National Measurement Institute

REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D887b.2013.01

Compound Name: **3,4-(Methylenedioxy)toluene**
Collection Number: D887b
Chemical Formula: $C_8H_8O_2$
CAS Registry Number: 62266-13-1
Description: Colourless liquid
Batch Number: 13-D-05
Molecular Weight: 136.1
Release date: 9th October 2013

Structure:



Synonyms: 5-Methyl-1,3-benzodioxole

Purity: 99.4% by GC-FID

This material is recommended for qualitative analysis only.

The purity value was obtained from GC-FID. Supporting evidence is provided by ^{1}H NMR and elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800	
	Column:	TG-17MS, 29.9 m × 0.32 mm I.D. × 0.25 µm	
	Program:	60 °C (1 min), 5 °C/min to 150 °C, 30 °C/min to 300 °C (5 min)	
	Injector:	250 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative peak area response of main component:		
	Initial analysis:	Mean = 99.4%, s = 0.01% (10 sub samples in duplicate, February 2013)	

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973	
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm	
	Program:	60 °C (1 min), 5 °C/min to 150 °C, 30 °C/min to 300 °C (3 min)	
	Injector:	250 °C	Transfer line temp: 280 °C
	Carrier:	Helium, 1.0 mL/min	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.

Parent (9.02 min): 135 (100), 78 (21), 77(20), 52 (10), 51 (14) m/z

IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	2886, 1491, 1437, 1344, 1246, 1192, 1042, 936, 800, 755 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance-400
	Field strength:	400 MHz Solvent: $CDCl_3$ (7.26 ppm)
	Spectral data:	δ 2.28 (3H, s), 5.91 (2H, s), 6.62 (1H, m), 6.67 (1H, m), 6.71 (1H, d, *J* = 8.0, Hz) ppm
^{13}C NMR:	Instrument:	Bruker Gyro-300
	Field strength:	75 MHz Solvent: $CDCl_3$ (77.2 ppm)
	Spectral data:	δ 21.1, 100.7, 108.0, 109.6, 121.5, 131.5, 145.3, 147.5 ppm
Microanalysis:		Found: C = 70.8%; H = 6.0% (March, 2013)
		Calc: C = 70.6%; H = 5.9% (Calculated for $C_8H_8O_2$)

Expiration of certification
The property values are valid till 25th February 2016, i.e. three years from the date of 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 been given a shelf life of three years from the date of certification. The material will be re-tested on an annual basis to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

In the absence of stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated from stability trials conducted on similar materials by NMI Australia over the last 10 years.

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 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: 9 October 2013

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