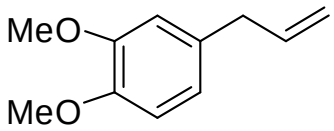
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D885: 1,2-Dimethoxy-4-(2-propenyl)benzene

Report ID: D885.2022.01

Chemical Formula: $C_{11}H_{14}O_2$

Molecular Weight: 178.2 g/mol



Property value

Batch No.	CAS No.	Purity (mass fraction)
04-D-23	**93-15-2**	**94.6 ± 3.2%**

IUPAC name: 1-(3,4-Dimethoxyphenyl)-2-propene

Expiration of certification: The property values are valid till 19 July 2025, i.e. three 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: Clear oil sourced from an external supplier, 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 and is not intended for use as a calibrator. The material does not have certified reference material status as metrological traceability of the stated purity value to the SI unit for mass (kg) has not been established.

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 4 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 ten 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.
21 July 2022

This report supersedes any issued prior to 22 July 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. The certified purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR spectroscopy. 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 elemental microanalysis.

Warning: **This material has shown significant signs of degradation when analysed as a 2000 μg/mL solution in chloroform. This suggests that this material is sensitive to the quality of the silanised glass liner and/or undergoes decomposition in chloroform.**

GC-FID:	Instrument:	Agilent 6890N or 8890
	Column:	HP-1, 30 m x 0.32 mm I.D. x 0.25 μm
	Program:	100 °C (1 min), 10 °C/min to 160 °C, 20 °C/min to 300 °C (3 min)
	Injector:	180 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.4%, s = 0.01% (10 replicates, March 2005)
	Re-analysis:	Mean = 97.0% s = 0.01% (3 sub samples in duplicate, July 2008)
	Re-analysis:	Mean = 95.3%, s = 0.004% (5 sub samples in duplicate, July 2022)
GC-FID:	Instrument:	Varian CP3800
	Column:	VF-1MS, 30 m x 0.32 mm I.D. x 0.25 μm
	Program:	100 °C (1 min), 10 °C/min to 160 °C, 20 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 95.3%, s = 0.07% (5 sub samples in duplicate, August 2011)
	Initial analysis:	Mean = 95.1%, s = 0.07% (5 sub samples in duplicate, February 2015)
Thermogravimetric analysis:		Non volatile residue < 0.2% mass fraction. Volatile content not determined due to volatility of the material.
Karl Fischer analysis:		Moisture content is 0.2% mass fraction (2 sub samples, June 2007) Moisture content is 0.3% mass fraction (2 sub samples, June 2008) Moisture content is 0.7% mass fraction (2 sub samples, July 2011) Moisture content is 0.8% mass fraction (2 sub samples, March 2015) Moisture content is 0.9% mass fraction (2 sub samples, June 2022)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	ZB-5, 30 m × 0.25 mm I.D. × 0.30 μm
	Program:	100 °C (1 min), 10 °C/min to 300 °C (1 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.	
	7.3 min:	178 (M^+, 100), 163 (26), 147 (28), 107 (20), 103 (24), 91 (26), 77 (12) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. 100% Chloroform Single spot observed, R_f = 0.6. Visualisation with UV at 254 nm.
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1}, KBr pellet
	Peaks:	3076, 3000, 2936, 2834, 1591, 1518, 1465, 1263, 1237, 1154, 1141, 1030, 914, 807 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 3.34 (2H, d, *J* = 6.7 Hz), 3.86 (3H, s), 3.87 (3H, s), 5.07 (1H, s), 5.08 (1H, d, *J* = 14 Hz), 5.96 (1H, m), 6.71-6.74 (2H, m), 6.80 (1H, d, *J* = 8.1 Hz) ppm
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	126 MHz
	Solvent:	$CDCl_3$ (76.9 ppm)
	Spectral data:	δ 39.7, 55.7, 55.9, 111.2, 111.8, 115.5, 120.3, 132.6, 137.6, 147.3, 148.8 ppm
Microanalysis:	Found:	C = 74.3%, H = 7.9% (April 2005)
	Calculated:	C = 74.1%, H = 7.9% (Calculated for $C_{11}H_{14}O_2$)