Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

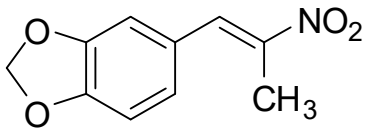
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D669: 1-(3,4-Methylenedioxyphenyl)-2-nitro-propene

Report ID: D669.2024.01

Chemical Formula: $C_{10}H_9NO_4$

Molecular Weight: 207.2 g/mol



Property value

Batch No.	CAS No.	Purity estimate
01-D-01	5438-41-5	99.7 ± 1.3%

IUPAC name: 5-[(1E)-2-Nitro-1-propen-1-yl]-1,3-benzodioxole.

Expiration of certification: The property values are valid till 4 September 2034, 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: Yellow needle prepared by synthesis, 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 should be used 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 4 ºC in a closed container in a dry, dark area.

Stability: This material has demonstrated stability over a minimum period of three years. This purity reported is an estimate of the E (trans) isomer content only. The Z (cis) isomer is also present, and interconversion of the E-Z isomers occurs in solution. For this reason the material is only recommended for use in qualitative or semi-quantitative studies.

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.
18 September 2024

This report supersedes any issued prior to 18 September 2024.

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 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.

This purity reported is an estimate of the E (*trans*) isomer content only. The Z (*cis*) isomer is also present, and interconversion of the E-Z isomers occurs in solution. For this reason the material is recommended for use in qualitative or semi-quantitative studies only.

GC-FID:	Instrument:	HP5890
	Column:	ZB-1 Capillary, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 10 °C/min to 250 °C (4 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 97.9%, s = 0.48% (10 samples in duplicate, February 2001)
	Re-analysis:	Mean = 99.1%, s = 0.19% (5 samples in duplicate, February 2006)
GC-FID:	Instrument:	Varian 3800
	Column:	VF-1MS Capillary, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	120 °C (1 min), 10 °C/min to 180 °C (1 min), 30 °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 = 99.2%, s = 0.1% (5 sub samples in duplicate, January 2011)
	Re-analysis:	Mean = 99.0%, s = 0.03% (6 sub samples in duplicate, December 2015)
	Re-analysis:	Mean = 99.4%, s = 0.05% (5 sub samples in duplicate, September 2020)
	Re-analysis:	Mean = 99.7%, s = 0.06% (5 sub samples in duplicate, September 2024)
Thermogravimetric analysis:		Volatile content was not measurable due to sublimation of the material Non-volatile residue < 0.2 % mass fraction (January 2001)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (February 2011,& December 2015) Moisture content 0.3% mass fraction (September 2020) Moisture content < 0.1% mass fraction (September 2024)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP 6890/5973
	Column:	Zebron ZB-5, 30 m × 0.25 mm I.D. × 0.30 μm
	Program:	100 °C (1 min), 12 °C/min to 280 °C, hold (4 min)
	Injector:	250°C
	Transfer line temp:	250 °C
	Carrier:	Helium
	Split ratio:	20/1
	The retention time of the E-isomer is reported along with the major peaks in the mass spectrum. The latter are reported in m/z. and (in brackets) as a percentage relative to the base peak.	
	7.3min:	207 (M^+, 74), 160 (78), 149 (18), 103 (100), 77 (48), 51 (18) *m/z*
	Instrument:	Varian Saturn 3400/2000 GC-MS Ion Trap
	Column:	HP Ultra 1, 22 m × 0.20 mm I.D. × 0.11 μm
	Program:	80 °C (1 min), 10 °C /min to 200 °C, 50 °C/min to 300 °C (3 min)
	Injector:	260 °C
	Transfer line temp:	300 °C
	Carrier:	Helium, 1 mL/min
	Split ratio:	40/1
	The retention time of the E-isomer is reported along with the major peaks in the mass spectrum. The latter are reported in m/z. and (in brackets) as a percentage relative to the base peak.	
	10.6 min:	207 (M^+, 39), 160 (72), 103 (67), 77 (100) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/hexane (1:1) Single spot observed, R_f = 0.3. Detection by visual inspection and UV light (254 nm)
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	1646, 1603, 1510, 1325, 1275, 1039 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Peaks:	δ 2.46 (3H, s), 6.05 (2H, s), 6.89(1H, d), 6.94 (1H, d), 6.98 (1H, dd), 8.02 (1H, s) ppm
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	126 MHz
	Solvent:	$CDCl_3$ (77.2 ppm)
	Peaks:	δ 14.2, 101.8, 108.9, 109.5, 126.0, 126.2, 133.6, 146.2, 148.2, 149.3 ppm
Melting point:		93-96 °C
Microanalysis:	Found:	C = 58.2%; H = 4.4%; N = 6.9% (February, 2001)
	Calculated:	C = 57.9%; H = 4.4%; N = 6.8% (Calculated for $C_{10}H_9NO_4$)