Australian Government
Department of Industry, Science and Resources

National Measurement Institute

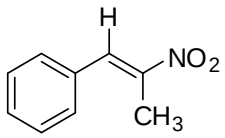
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D869: 1-Phenyl-2-nitro-propene

Report ID: D869.2020.03

Chemical Formula: $C_9H_9NO_2$

Molecular Weight: 163.2 g/mol



Purity value

Batch No.	CAS No.	Purity estimate
04-D-11	**705-60-2**	**99.2 ± 1.2%**

IUPAC name: [(1E)-2-Nitro-1-propen-1-yl] benzene

Expiration of certification: The property values are valid till 9 April 2030, 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 solid prepared by synthesis, 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 25 ºC in a closed container in a dry, dark area.

Stability: This material has demonstrated stability over a minimum period of ten 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.

Caution: 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.
13 October 2022

This report supersedes any issued prior to 19 September 2022.

NATA Accreditation No. 198 / Corporate Site No. 14214.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. 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.

GC-FID:	Instrument:	Varian CP-3800
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	60 °C (1 min), 15 °C/min to 250 °C (1 min), 20 °C/min to 300 °C (5 min)
	Injector:	180 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.8%, s = 0.06% (10 sub samples in duplicate, June 2004)
	Re-analysis:	Mean = 99.5%, s = 0.02% (5 sub-samples in duplicate, September 2008)
	Re-analysis:	Mean = 99.6%, s = 0.08% (5 sub-samples in duplicate, April 2020)
GC-FID:	Instrument:	Varian CP-3800
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	60 °C (1 min), 15 °C/min to 250 °C (1 min), 20 °C/min to 300 °C (2 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.4%, s = 0.03% (5 sub samples in duplicate, September 2011)
GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-5, 30.0 m × 0.32 mm I.D. × 0.25 µm
	Program:	60 °C (1 min), 15 °C/min to 250 °C (1 min), 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 = 99.1%, s = 0.05% (7 sub samples in duplicate, June 2016)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (October 2008, September 2011) Moisture content ca 0.13% mass fraction (June 2016) Moisture content ca 0.16% mass fraction (April 2020)
Thermogravimetric analysis:		Non-volatile residue < 0.2 % total mass fraction. Volatiles not determined due to volatility of material

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP 6890/5973
	Column:	ZB-5, 30 m × 0.25 mm I.D. × 0.25 µm
	Program:	60 °C (1 min), 10 °C/min to 250 °C
	Injector:	220 °C
	Transfer line temp:	280 °C
	Carrier:	Helium
	Split ratio:	20/1
	The retention time of the major isomer 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.	
	13.2 min:	163 (M^+, 13), 146 (10), 115 (100), 105 (31), 91 (37), 77 (11), 63 (10), 51 (11) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/hexane (1/1) Single spot observed, R_f = 0.4
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400cm^{-1}, KBr pellet
	Peaks:	3057, 2975, 2809, 2417, 2182, 1972, 1651, 1514, 1321, 1216, 980, 764 cm^{-1}
^{1}H NMR:	Instrument:	Gyro-300
	Field strength:	300 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 2.46 (3H, d, *J* = 0.8 Hz), 7.30-7.60 (5H, m), 8.09 (1H, s) ppm
^{13}C NMR:	Instrument:	Gyro-300
	Field strength:	75 MHz
	Solvent:	$CDCl_3$ (77 ppm)
	Spectral data:	δ 14.0, 125.9, 128.9, 129.9, 132.4, 133.5, 147.8 ppm
Melting point:		63 - 64 °C
Microanalysis:	Found:	C = 66.4%; H = 5.6%; N = 8.7% (July 2004) C = 66.4%; H = 5.5%; N = 8.8% (August 2005)
	Calculated:	C = 66.3%; H = 5.6%; N = 8.6% (Calculated for $C_9H_9NO_2$)