Australian Government
Department of Industry, Science and Resources

National Measurement Institute

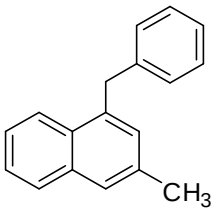
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D456: 1-Benzyl-3-methylnaphthalene

Report ID: D456.2021.03

Chemical Formula: $C_{18}H_{16}$

Molecular Weight: 232.3 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
95-056556	**93870-53-2**	**99.6 ± 0.3%**

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit (k = 2).

Synonyms: 1-Benzyl-3-methyl-naphthalin

Expiration of certification: The property values are valid till 19 February 2031, 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. In the event a product fails the stability trial, notification will be sent to all impacted customers.

Description: White crystal prepared by synthesis or 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 should be used for qualitative analysis only.

Instructions for use: This certified reference material is suitable for use as a primary calibrator.

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.

Metrological traceability: The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance approach all impurities are quantified as a mass fraction and subtracted from 100%.

Stability: This material has demonstrated stability over a minimum period of ten years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials. 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 five 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.

Safety: Treat as 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.
14 September 2022

This report supersedes any issued prior to 14 September 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 purity estimate was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, Karl Fischer analysis and ^{1}H NMR spectroscopy. The purity value is calculated as per Equation 1.

$$\text{Purity} = (100\ \% - I_{ORG}) \times (100\ \% - I_{VOL} - I_{NVR}) \qquad \text{Equation 1}$$

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue

GC/FID	Instrument:	Agilent 6890N
	Column:	HP-1, 30 m x 0.32 mm I.D. x 0.25 μm
	Program:	180 °C (1 min), 10 C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector:	320 °C
	Carrier:	Helium
	Split rate:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.10% (3 sub samples in duplicate, April 1996)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, May 2007)
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, May 2011)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, April 2016)
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, February 2021)
Karl Fischer analysis:		Moisture content 0.8% mass fraction (2 sub samples, May 2007) Moisture content 0.5% mass fraction (3 sub samples, May 2008) Moisture content ≤ 0.2% mass fraction (7 sub samples, May 2011; 2 sub samples, April 2016 and March 2021)

Spectroscopic and other characterisation data

GC/MS:	Instrument:	HP5890/5970B
	Column:	HP Ultra-2, 12 m x 0.22 mm I.D. x 0.11 μm film thickness
	Temp program:	70 °C to 300 °C at 10 °C/min
	Scan Range:	40-450 *m/z*
	Ionisation:	EI/70 eV
	Injector Temp:	230 °C
	Transfer Line temp:	280 °C
	Carrier Gas:	Helium 1 mL/min
	Split ratio:	10/1
	Peaks:	232, 217, 215, 202, 189, 165, 155, 139, 128, 115, 108, 101, 91 *m/z* Matches literature reference spectrum.
HPLC:	Relative mass fraction of the main component, mean of replicates = 99.5%, s = 0.2%	
	Column:	Alltima C-18, 5 μm x 4.6 mm x 250 mm
	Mobile Phase:	Acetonitrile/water (85/15); Rt = 9.9 min
	Flow Rate:	1.0 mL/min
	Wavelength:	254 nm
TLC:	Conditions:	Kieselgel 60 F254 plate. Hexane (100%) Single spot observed R_f = 0.2 (Impurities < 0.2%)
IR:	Instrument:	FT-IR, BIORAD WIN FTS40
	Range:	4000-400 cm^{-1}. KBr pellet
	Peaks:	1601, 1494, 1452, 875, 850, 746, 726, 698 cm^{-1}
^{1}H NMR:	Instrument:	VARIAN GEMINI
	Field strength	300 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data	δ 2.48 (s, 3H), 4.42 (s, 2H), 7.1-7.3 (m, 5H), 7.3-7.5 (m, 2H), 7.53(s, 1H), 7.7-7.8 (m, 1H), 7.9-8.0 (m, 1H) ppm Matches literature reference spectra
^{13}C NMR:	Instrument:	VARIAN GEMINI
	Field strength	75 MHz
	Solvent:	$CDCl_3$ (77.16 ppm)
	Spectral data	δ 21.7, 39.0, 124.1, 125.1, 125.6, 126.1, 126.1, 128.0, 128.4, 128.7, 129.7, 130.5, 134.3, 135.0, 136.4, 140.7 ppm
Melting point:		60-61 °C