Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

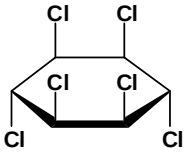
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1332: Lindane

Report ID: P1332.2023.01

Chemical Formula: $C_6H_6Cl_6$

Molecular Weight: 290.8 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
96-034442	**58-89-9**	**99.7 ± 0.4%**

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

IUPAC name: ɣ-1,2,3,4,5,6-Hexachlorocyclohexane

Expiration of certification: The property values are valid till 4 July 2028, five 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: White crystals 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 certified reference material is suitable for use as a primary calibrator.

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.

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 five 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 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.
5 July 2023

This report supersedes any issued prior to 5 July 2023.

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, 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 qualitative elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	120 °C (1 min), 15 °C/min to 300 °C (3 min)
	Injector:	230 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.1% (7 sub samples in duplicate, June 1996)
	Re-analysis:	Mean = 99.9%, s = 0.003% (5 sub samples in duplicate, December 2008)
	Re-analysis:	Mean = 99.9%, s = 0.005% (5 sub samples in duplicate, October 2013)
	Re-analysis:	Mean = 99.9%, s = 0.002% (5 sub samples in duplicate, April 2019)
	Re-analysis:	Mean = 99.8%, s = 0.006% (5 sub samples in duplicate, July 2023)
HPLC:	Column:	Alltima C-18 5 μm (4.6 mm x 150 mm)
	Mobile Phase:	Acetonitrile/water (88/12)
	Flow Rate:	0.8 mL/min
	Detector:	Refractive Index
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.1% (3 sub samples, June 1996)
Karl Fischer analysis:		Moisture content ≤ 0.1% mass fraction (December 2008 - June 2023)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP5890/5970B
	Ionisation:	NICI
	Column:	HP Ultra-2, 12 m x 0.22 mm I.D. x 0.11 μm
	Program:	70 °C to 300 °C at 10 °C/min
	Injector:	230 °C
	Split ratio:	10/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	35-450 *m/z*
	Peaks observed at 255, 71, 35 *m/z*. Matches literature reference spectrum	
TLC:	Conditions:	Kieselgel 60F_{254}. cyclohexane/diisopropyl ether/diethylamine, 52:40:8 Single spot observed, R_f = 0.5
IR:	Instrument:	FT-IR, BIORAD WIN FTS40
	Range:	4000-400 cm^{-1}, KBr pellet
	Peaks:	1342, 1100, 953, 911, 851, 779, 686, 480, 416 cm^{-1}
	The IR spectrum obtained for P1332 matches literature reference spectrum.	
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 4.65 (4H, d, *J* = 5.0 Hz), 4.76 (2H, bs) ppm
		^{1}H NMR shows the presence of hexane and methanol in quantities of 0.01% and 0.07% mass fractions respectively.
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	$CDCl_3$ (77 ppm)
	Spectral data:	δ 60.2, 60.6 ppm
Melting point:		111.5-112.5 °C
Microanalysis:	Found:	C = 24.96%; H =2.05% (December, 2005)
	Calculated:	C = 24.78%; H =2.08 % (Calculated for $C_6H_6Cl_6$)