Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

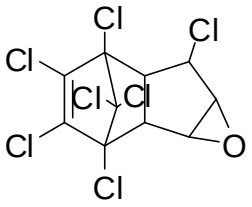
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1395: Heptachlor epoxide (exo)

Report ID: P1395.2023.01

Chemical Formula: $C_{10}H_5Cl_7O$

Molecular Weight: 389.3 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
96-104158	**1024-57-3**	**99.6 ± 0.3%**

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

IUPAC name: (2R,3S,5S,6R,7S)-1,6,8,9,10,11,11-Heptachloro-4-oxatetracyclo[6.2.1.02,7.03,5]undec-9-ene.

Expiration of certification: The property values are valid till 10 March 2033, 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: White platelet 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 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 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.
22 March 2023

This report supersedes any issued prior to 22 March 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, 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:	Agilent 6890N or 8890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	120 °C (3 min), 15 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 100%, s = 0.00% (3 sub samples, December 1997)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, February 2009)
	Re-analysis:	Mean = 99.9%, s = 0.02% (5 sub samples in duplicate, December 2013)
	Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, November 2018)
	Re-analysis:	Mean = 99.9%, s = 0.04% (5 sub samples in duplicate, March 2023)
Karl Fischer analysis:		Moisture content ≤ 0.2% mass fraction (February 2009, December 2013, November 2018 and January 2023)
Thermogravimetric analysis:		Volatile contents not determined due to the nature of the material (February 2009)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP6890/5973
	Column:	DB5-MS, 24 m × 0.25 mm I.D. × 0.25 µm
	Program:	70 °C (1 min), 10 °C/min to 130 °C, 20 °C/min to 310 °C (3 min)
	Injector:	230 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium
	Scan range:	50-550 *m/z*
	The retention time of the parent compound isreported with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (11.3 min):	390 (M^+, 10), 388 (M+, 10), 355 (87), 353 (100), 81 (81) *m/z*
HPLC:	Instrument:	LKB Bromma HPLC pump
	Column:	Alltima C-18 5 µm (4.6 mm × 250 mm)
	Mobile Phase:	Acetonitrile/MilliQ water (80:20)
	Flow rate:	1 mL/min
	Detector:	HP1047A Refractive index detector
	Relative peak area of main component:	
	Initial analysis:	Mean = 100%, s = 0.00% (7 sub samples in duplicate, November 1996)
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/Acetone (5:1) Single spot observed, R_f = 0.47. Visualised with iodoplatinate spray
IR:	Instrument:	Biorad WIN FTS40 FT-IR
	Range:	4000-400 cm^{-1}, KBR pellet
	Peaks:	2997, 1603, 1260, 1170, 1046, 912, 858, 679, 578, 454 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance 400
	Field strength:	400 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 3.21 (1H, dd, *J* = 3.0, 7.6 Hz), 3.79 (1H, d, *J* = 7.6 Hz), 3.90 (1H, d, *J* = 1.8 Hz), 4.02 (1H, t, *J* = 2.1 Hz), 4.34 (1H, dd, *J* = 1.2, 2.9) ppm
^{13}C NMR:	Instrument:	Bruker Avance 400
	Field strength:	100 MHz
	Solvent:	DMSO-d_6 (39.52 ppm)
	Spectral data:	δ 55.4, 56.6, 58.6, 60.6, 62.3, 78.8, 80.3, 103.0, 129.9, 131.0 ppm
Microanalysis:	Found:	C = 31.1%; H = 1.1% (November 2001)
	Found:	C = 30.8%; H = 1.2% (February 2009)
	Calculated:	C = 30.9%; H = 1.3% (Calculated for $C_{10}H_5Cl_7O$)