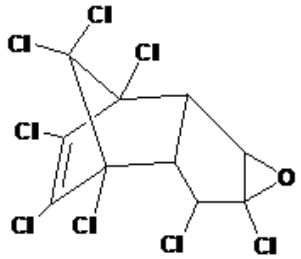
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA P1622: Oxychlordane



Report ID: P1622.2020.03 (Ampouled 110725)

Chemical Formula: $C_{10}H_4Cl_8O$

Molecular Weight: 423.8 g/mol

Property value

Batch No.	CAS No.	Mass per ampoule
96-106622	27304-13-8	989 µg

IUPAC name: 1,5,6,8,9,10,11,11-Octachloro-4-oxatetracyclo[6.2.1.$0^{2,7}$.$0^{3,5}$]undec-9-ene

Expiration of certification: The property values are valid till 30 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 ampoules that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials.

Description: The compound is supplied as a dried aliquot in a sealed ampoule under an atmosphere of argon. The reference material is intended for a single use to prepare a standard solution containing P1622. The material was prepared by synthesis, and certified for identity and purity by NMIA.

Intended use: This reference material should be used for qualitative analysis only.

Instructions for use: Open the ampoule and carefully rinse the interior at least three times with a suitable organic solvent (e.g. Chloroform). This will transfer 989 µg of anhydrous oxychlordane. The mass of analyte in each ampoule is calculated from the assigned purity of the bulk and the concentration of bulk material in a stock solution used to prepare the ampoules.

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 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 seven randomly selected ampoules 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 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.
6 September 2022

This report supersedes any issued prior to 6 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:

GC-FID:	Instrument:	Agilent 6890 or 7890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	130 °C (1 min), 10 °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 = 98.7%, s = 0.01% (7 ampoules in duplicate, July 2011)
	Re-analysis:	Mean = 98.7%, s = 0.02% (5 ampoules in duplicate, May 2012)
	Re-analysis:	Mean = 98.7%, s = 0.01% (5 ampoules in duplicate, May 2015)
	Re-analysis:	Mean = 98.6%, s = 0.02% (5 ampoules in duplicate, April 2020)

The following analytical data was obtained on the bulk material subsequently used in the preparation of the ampoules.

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:	Agilent 5890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	130 °C (1 min), 10 °C/min to 280 °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 = 98.8%, s = 0.04% (7 sub samples in duplicate, December 1999)
	Re-analysis:	Mean = 98.6%, s = 0.02% (8 sub samples in duplicate, May 2006)
GC-FID:	Instrument:	Agilent 7890
	Column:	HP-1MS, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	130 °C (1 min), 10 °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:	98.6%, s = 0.04% (5 sub samples in duplicate, June 2011)
Karl Fischer analysis:		Moisture content 0.1% mass fraction (2 sub samples, June 2011)
Thermogravimetric analysis:		Volatile content < 0.1% and non volatile residue < 0.2% mass fraction (August 1999 & May 2006)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890/5973
	Column:	ZB-5, 30 m × 0.25 mm I.D. × 0.25 µm
	Program:	160 °C (1 min), 15 °C/min to 320 °C (3 min)
	Injector:	250 °C
	Transfer line temp:	280 °C
	Carrier:	Helium
	Split ratio:	50/1
	The retention time of the parent compound is reported along with the main ^{35}Cl isomeric species in the mass spectrum. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (7.3 min):	387 (M^+-Cl, 48), 263 (31), 187 (66), 185 (71), 149(54), 117 (40), 115 (100) *m/z*
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	3500-500 cm^{-1}, KBr powder
	Peaks:	1605, 1388, 1285, 1252, 1171, 1105, 1067, 1013, 973, 945, 913, 825, 710, 688, 585, 510, 458 cm^{-1}
^1H NMR:	Instrument:	Bruker DMX-300
	Field strength:	300 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 3.35 (1H, ddd, *J* = 0.73, 2.8, 7.8 Hz), 3.55 (1H, d, *J* = 11.1 Hz), 3.93(1H, s), 4.37 (1H, d, *J* = 2.8 Hz) ppm
Melting point:		93-96 °C
Microanalysis:	Found:	C = 28.7 %; H = 0.8 %; Cl = 67.8% (November, 1999)
	Calculated:	C = 28.3 %; H = 1.0 %; Cl = 66.9% (Calculated for $C_{10}H_4Cl_8O$)