Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

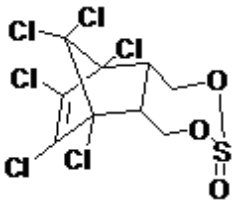
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1368: Endosulfan (alpha)

Report ID: P1368.2021.02

Chemical Formula: $C_9H_6Cl_6O_3S$

Molecular Weight: 406.9 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
96-104172	959-98-8	99.7 ± 0.3%

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

IUPAC name: (1R,2R,8S,9S)-1,9,10,11,12,12-Hexachloro-4,6-dioxa-5-thiatricyclo[7.2.1.02,8]dodec-10-ene 5-oxide.

Expiration of certification: The property values are valid till 12 October 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.

Description: Off-white powder sourced from an external supplier, and certified for identity and purity by NMIA. Packaged in amber glass bottles with a 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.
19 August 2022

This report supersedes any issued prior to 19 August 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 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 qualitative headspace GC-MS analysis of occluded solvents, ^{1}H NMR and elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890		
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm		
	Program:	180 °C (1 min), 10 °C/min to 260 °C (1 min), 40 °C/min to 300 °C (1 min)		
	Injector:	230 °C	Detector Temp:	320 °C
	Carrier:	Helium	Split ratio:	20/1
	Relative mass fraction of the main component:			
	Initial analysis:	Mean = 99.8%, s = 0.02% (3 sub samples in duplicate, October 2001)		
	Re-analysis:	Mean = 99.7%, s = 0.1% (3 sub samples in duplicate, October 2002)		
	Re-analysis:	Mean = 99.8%, s = 0.001% (5 sub samples in duplicate, February 2004)		
	Re-analysis:	Mean = 99.7%, s = 0.1% (5 sub samples in duplicate, February 2007)		
GC-FID:	Instrument:	Varian CP-3800		
	Column:	VF-1MS, 29.58 m × 0.32 mm I.D. × 0.25 μm		
	Program:	180 °C (1 min), 10 °C/min to 260 °C (1 min), 30 °C/min to 300 °C (3min)		
	Injector:	250 °C	Detector Temp:	320 °C
	Carrier:	Helium	Split ratio:	20/1
	Relative peak area response of main component:			
	Initial analysis:	Mean = 99.8%, s = 0.007% (5 sub samples in duplicate, January 2012)		
GC-FID:	Instrument:	Agilent 8890		
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm		
	Program:	180 °C (1 min), 10 °C/min to 260 °C (1 min), 30 °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 = 99.8%, s = 0.01% (5 sub samples in duplicate, October 2021)		
Karl Fischer analysis:		Moisture content ≤ 0.2% mass fraction (February 2007, January 2012 & September 2021)		
Thermogravimetric analysis:		Non-volatile residue < 0.2% mass fraction (June 2001)		

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP6890/5973
	Column:	HP Ultra-2, 12 m x 0.22 mm I.D. x 0.10 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
	Scan range:	50-550 *m/z*
	The retention time of the parent compound is reported 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 (4.1 min):	339 (44), 277 (57), 265 (62), 241 (96), 207 (83), 195 (100), 170 (77) *m/z*
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 μm
	Program:	50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min)
	Injector:	150 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Split ratio:	50/1
	Solvents detected:	Hexane
TLC:	Conditions:	Kieselgel 60F_{254}. Diisopropylether/diethylether/diethylamine (45/45/10) Single spot observed, R_f = 0.25
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400 cm^{-1}, KBr pellet
	Peaks:	1605, 1271, 1194, 1006, 980, 859, 792, 754, 701, 674, 623, 582 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 3.44 (2H, m, *J* = 10.1 Hz), 3.95 (2H, dd, *J* = 3.4, 3.4 Hz), 4.76 (2H, m, *J* = 12.0 Hz) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	$CDCl_3$ (77.2 ppm)
	Spectral data:	δ 51.3, 56.6, 79.7, 103.1, 131.1 ppm
Melting point:		106-108 °C
Microanalysis:	Found:	C = 26.6%; H = 1.5%; S = 7.6% (September, 2001)
	Calculated:	C = 26.6%; H = 1.5%; S = 7.9% (Calculated for $C_9H_6Cl_6O_3S$)