



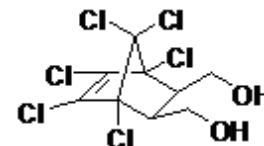
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1370: Endosulfan diol

Report ID: P1370.2018.04

Chemical Formula: C₉H₈Cl₆O₂

Molecular Weight: 360.9 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
96-104186	2157-19-9	99.6 ± 1.0%

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

IUPAC name: (1,4,5,6,7,7-Hexachlorobicyclo[2.2.1]hept-5-ene-2,3-diyl)dimethanol

Expiration of certification: The property values are valid till 31 January 2028, 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: Pale yellow 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 °C 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%. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

Stability: This material has demonstrated stability over a minimum period of 10 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 August 2022

This report supersedes any issued prior to 22 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 from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (qNMR). The techniques used in the mass balance approach include GC-FID, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR spectroscopy. The purity value is calculated as per Equation 1.

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

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

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified purity value by qNMR was obtained using the one-proton doublet of doublets at 5.1 ppm measured against a certified internal standard of dimethyl terephthalate.

Supporting evidence is provided by qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument:	HP5890	
	Column:	ZB-1 capillary, 29.5 m x 0.32 mm I.D. 0.25 µm	
	Program:	180 °C (2 min), 10 °C/min to 260 °C (5 min)	
	Injector:	230 °C	Detector Temp: 315 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative mass fraction of the main component:		
	Initial analysis:	Mean = 99.7%, s = 0.3% (1996)	
	Re-analysis:	Mean = 99.9%, s = 0.0% (5 sub samples in duplicate, April 2007)	
		Varian 3800	
	Column:	HP-1 capillary, 29.5 m x 0.32 mm I.D. 0.25 µm	
	Program:	180 °C (1 min), 6 °C/min to 220 °C (5 min), 30 °C/min to 280 °C (3 min)	
	Injector:	250 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative mass fraction of the main component <i>bis</i> -TMS derivative:		
	Initial analysis:	Mean = 99.9%, s = 0.009% (June 2010)	
	Re-analysis:	Mean = 99.9%, s = 0.003% (5 sub samples in duplicate, January 2018)	
		Column: Alltima C-18 5 µm (4.6 mm x 250 mm)	
	Mobile Phase:	Acetonitrile/water (80/20)	
	Flow Rate:	1.0 mL/min	Detector: Refractive Index detector
	Relative peak area response of main component:		
	Initial analysis:	Mean = 99.7%, s = 0.4% (1996)	
Karl Fischer analysis:	Moisture content < 0.1% mass fraction (May 2010 and February 2018)		
Thermogravimetric analysis:	Volatile content < 0.1% and non-volatile content < 0.2% mass fraction (April 2007)		
QNMR:	Instrument:	Bruker Avance-III-500	
	Field strength:	500 MHz	Solvent: DMSO (2.54 ppm)
	Internal standard:	Dimethyl terephthalate (100.0% mass fraction)	
	Initial analysis:	Mean (5.1 ppm) = 99.3%, s = 0.3% (5 sub samples, October 2010)	

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP5890/5970
	Column:	HP Ultra-2, 12 m x 0.22 mm I.D. Film thickness 0.11 μm
	Program:	70 $^{\circ}\text{C}$ to 300 $^{\circ}\text{C}$ at 20 $^{\circ}\text{C}/\text{min}$
	Injector:	230 $^{\circ}\text{C}$
	Split ratio:	10/1
	Transfer line temp:	280 $^{\circ}\text{C}$
	Carrier:	Helium
	Scan range:	40-450 m/z
	The retention time of <i>bis</i> -TMS derivative 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. The mass spectrum of the parent compound matches a reference spectrum for endosulfan diol	
	Parent:	362, 361, 342, 307, 277, 259, 229, 193, 143, 119, 85, 69 m/z
	<i>Bis</i> -TMS:	433, 379, 330, 271, 231, 177, 147, 103, 73 m/z
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Cyclohexane/Diisopropylether/Diethylamine (52/40/8) Single spot observed, $R_f = 0.68$
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1} , KBr pellet
	Peaks:	1603, 1264, 1166, 1096, 1077, 1042, 1010, 913, 853, 792, 697, 600 cm^{-1}
¹ H NMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz
	Solvent:	MeOH- <i>d</i> ₄ (3.34 ppm)
	Spectral data:	δ 3.18 (2H, m), 3.66 (2H, m), 3.83 (2H, m) ppm
¹³ C NMR:	Instrument:	Bruker DMX-600
	Field strength:	151 MHz
	Solvent:	MeOH- <i>d</i> ₄ (49.9 ppm)
	Spectral data:	δ 51.8, 58.0, 81.0, 103.0, 131.1 ppm
Melting point:		207-210 $^{\circ}\text{C}$
Microanalysis:	Found:	C = 30.1%; H = 2.4% (May, 2007)
	Calculated:	C = 30.0%; H = 2.2% (Calculated for C ₉ H ₈ Cl ₆ O ₂)