Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

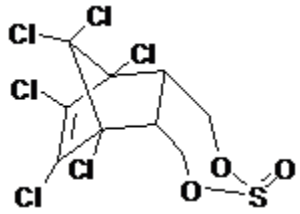
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1369: Endosulfan (beta)

Report ID: P1369.2021.02

Chemical Formula: $C_9H_6Cl_6O_3S$

Molecular Weight: 406.9 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
96-104183	33213-65-9	99.3 ± 0.8%

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 6 August 2026, i.e. 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 sourced from an external supplier, 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%. 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 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.
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 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 ^{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

The purity value by qNMR was obtained using a combination of the two-proton doublet at 4.1 ppm, the two-proton doublet at 5.1 ppm and the one-proton multiplet at 4.6 ppm measured against a certified internal standard of triphenylphosphine oxide.

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

GC-FID:	Instrument:	Agilent 5890N or 6890
	Column:	HP-1 MS, 30 m x 0.32 mm I.D.x 0.25 µm
	Program:	180 °C (2 min), 10 °C/min to 260 °C (1 min)
	Injector:	230 °C
	Detector Temp:	315 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.05% (3 samples in duplicate, September 1996)
	Re-analysis:	Mean = 99.7%, s = 0.01% (3 sub samples in duplicate, January, 2004)
	Re-analysis:	Mean = 99.8%, s = 0.003% (5 sub samples in duplicate, July, 2007)
GC-FID:	Instrument:	Agilent 6890N or 8890
	Column:	HP-1 MS, 30 m x 0.32 mm I.D.x 0.25 µm
	Program:	180 °C (2 min), 10 °C /min to 260 °C (1 min), 20 °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 = 99.8%, s = 0.004% (7 sub samples in duplicate, November 2011)
	Re-analysis:	Mean = 99.8%, s = 0.09% (5 sub samples in duplicate, October 2016)
	Re-analysis:	Mean = 99.8%, s = 0.006% (5 sub samples in duplicate, August 2021)
HPLC:	Instrument:	Shimadzu
	Column:	Alltima C-18, 2.7 µm (4.6 mm x 150 mm)
	Mobile Phase:	Acetonitrile/MilliQ water (80:20)
	Flow rate:	1.0 mL/min
	Detector:	Refractive Index
	Relative peak area response of main component:	
	Initial analysis:	Mean = 99.9%, s = 0.1% (7 sub samples, September 1996)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (April 2015) Moisture content < 0.1% mass fraction (June 2021)
Thermogravimetric analysis:		Volatiles content < 0.1% and non-volatile content < 0.2% mass fraction (October 2001 and July 2007)
QNMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz Solvent: DMSO-d_6 (2.50 ppm)
	Internal standard:	Triphenylphosphine oxide (100% mass fraction)
	Initial analysis:	Mean (4.1 ppm) = 99.3%, s = 0.3% (5 sub samples in duplicate, Nov 2011) Mean (5.1 ppm) = 98.3%, s = 0.4% (5 sub samples in duplicate, Nov 2011)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP5890/5989A
	Column:	DB-5MS, 24 m x 0.25 mm I.D. Film thickness 0.11 μm
	Program:	70 °C to 300 °C at 20 °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 (4.8 min):	406 (M^+, 6), 338 (47), 276 (46), 264 (55), 236 (98), 206 (80), 194 (100) *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}. Cyclohexane/diisopropylether/diethylamine (52/40/8) Single spot observed, R_f = 0.76
IR:	Instrument:	FT-IR, BIORAD WIN FTS40
	Range:	4000-400 cm^{-1}, KBr pellet
	Peaks:	1607, 1249, 1194, 1146, 990, 959, 913, 881, 779, 676, 633, 480 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 3.15 (2H, s), 4.12 (2H, d, *J* = 14.5 Hz), 5.08 (2H, d, *J* = 13.6 Hz) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	$CDCl_3$ (77.16 ppm)
	Spectral data:	δ 52.4, 55.1, 81.0, 103.6, 131.2 ppm
Melting point:		209-210 °C
Microanalysis:	Found:	C = 26.6%; H = 1.5%; S = 7.4% (September 2001)
	Found:	C = 26.8%; H = 1.4%; S = not determined (November 2005)
	Calculated:	C = 26.6%; H = 1.5%; S = 7.9% (Calculated for $C_9H_6Cl_6O_3S$)