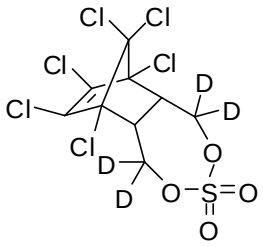
Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

DEUTERATED INTERNAL STANDARD PRODUCT INFORMATION SHEET

NMIA P1803: d4- Endosulfan sulfate



Report ID: P1803.2016.02

Chemical Formula: $C_9H_2D_4Cl_6O_4S$

Molecular Weight: 427.0 g/mol

Property value

Batch No.	CAS No.	Purity estimate
06-AV-01	**Not applicable**	**97.3%**

IUPAC name: d_4-(1R,2R,8S,9S)-1,9,10,11,12,12-Hexachloro-4,6-dioxa-5-thiatricyclo[7.2.1.02,8]dodec-10-ene 5,5-dioxide

Expiration of certification: The property values are valid till 23 May 2021, 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 powder 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: The isotopic purity of this material is an estimate only. This material should be considered for use as an internal standard only.

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.

Stability: 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.
9 April 2020

This report supersedes any issued prior to 9 April 2020

NATA logo notice: Accredited for compliance with ISO 17034. Accreditation No. 198 / Corporate Site No. 20844. The results of the tests, calibrations and/or measurements included in this document are traceable to Australian/national standards.

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 purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, 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 and elemental microanalysis.

The main component of this material is d_4-endosulfan sulfate. d_3-, d_2-, d_1- and d_0- endosulfan sulfate are also present. The stated chemical purity of the analyte represents the combined mass fractions of deuterated (d_4, d_3, d_2 and d_1) and d_0- endosulfan sulfate in the material.

The isotopic purity of this material is an estimate only. This material should be considered for use as an internal standard only.

Isotopic Purity: $d_4 \approx 93\%$ [= $d_4/(d_4 + d_3 + d_2 + d_1 + d_0)$ x 100]

$d_0 \approx 0\%$ [= $d_0/(d_4 + d_3 + d_2 + d_1 + d_0)$ x 100]

(Determined by SIM analysis)

Note: 1 mg of the material contains approximately 904 µg of d_4-endosulfan sulfate.

GC-FID:	Instrument:	Agilent 6890N
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	180 °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 = 97.4%, s = 0.06% (7 sub samples in duplicate, June 2006)
	Re-analysis:	Mean = 97.4%, s = 0.02% (5 sub samples in duplicate, July 2007)
	Re-analysis:	Mean = 97.4%, s = 0.01% (5 sub samples in duplicate, August 2008)
	Re-analysis:	Mean = 97.5%, s = 0.04% (5 sub samples in duplicate, August 2011)
	Re-analysis:	Mean = 97.5%, s = 0.04% (5 sub samples in duplicate, May 2016)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (July 2006, July 2007, July 2008 and July 2011) Moisture content ca 0.1% mass fraction (June 2016)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP6890/5973
	Column:	ZB-5ms, 26 m × 0.25 mm I.D. × 0.25 µm
	Program:	70 °C (1 min), 20 °C/min to 300 °C (2 min)
	Injector:	230 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	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 (11.4 min): 428 (9), 426 (14), 424 (6), 393 (29), 391 (52), 389 (24), 274 (92), 272 (100), 270 (67), 237 (67), 231 (68), 229 (60), 207 (25), 172 (24), 141 (18), 103 (17) *m/z*

This material was shown to co-elute with a sample of native endosulfan sulfate (NMI Collection Number: P1372)

TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/ethyl acetate (4:1) Single spot observed, R_f = 0.35 Visualisation with potassium permanganate.
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	2992, 2924, 2176, 2128, 1605, 1405, 1391, 1216, 1173, 1008, 902, 767 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 3.25 (0.32H, bs), 3.61 (1.68H, bs) ppm (two conformational isomers) Dichloromethane, estimated at 0.13% mass fraction, observed at δ 5.30 ppm
^{13}C NMR:	Instrument:	Bruker Gyro-300
	Field strength:	75 MHz
	Solvent:	$CDCl_3$ (77.2 ppm)
	Spectral data:	δ 49.5 ppm. (other peaks not visible due to deuteration and peak broadening, caused by conformational isomers)
	Melting point:	141-145 °C
Microanalysis:	Found:	C = 25.4 %; H = 0.5 %; D = 1.9; S = 7.3% (June 2006)
	Calculated:	C = 25.3 %; H = 0.5 %; D = 1.9; S = 7.5% (Calculated for $C_9H_2D_4Cl_6O_4S$)