



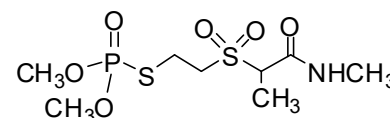
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1730: Vamidothion sulfone

Report ID: P1730.2025.01 (Bottled 160413)

Chemical Formula: $C_8H_{18}NO_6PS_2$

Molecular Weight: 319.3 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
01-AV-05	70898-34-9	99.6 ± 0.5%

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

IUPAC name: O,O-Dimethyl S-(2-[[1-(methylamino)-1-oxo-2-propenyl]sulfonyl]ethyl) phosphorothioate.

Expiration of certification: The property values are valid till 14 July 2035, 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: White solid sourced from an external supplier and certified for identity and purity by NMI Australia. 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 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 HPLC with UV detection 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.
10 September 2025

This report supersedes any issued prior to 10 September 2025.

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 HPLC with CAD detection, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR spectroscopy. The purity value is calculated as per Equation 1.

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

Equation 1

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

Supporting evidence is provided by quantitative NMR using certified triphenylphosphine oxide as internal standard, and elemental microanalysis.

microanalysis.

HPLC:	Instrument:	Thermo Scientific UltiMate 3000
	Column:	X-Bridge C-18, 5 µm (4.6 mm × 250 mm)
	Column oven:	40 °C
	Mobile Phase:	Methanol/Milli-Q water (25:75 v/v)
	Flow rate:	1.0 mL/min
	Detector:	Corona Ultra RS CAD detector
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, August 2021)
	Re-analysis:	Mean = 99.6%, s = 0.02% (5 sub samples in duplicate, July 2025)
HPLC:	Instrument:	Waters HPLC 1525 pump, 717 autosampler
	Column:	Alltima C-18 5 µm (4.6 mm × 150 mm)
	Mobile Phase:	A: Milli-Q water, B: Methanol (75% A, 25% B)
	Flow Rate:	1.0 mL/min
	Detector:	ELSD
	Relative area response of the main component:	
	Initial analysis:	Mean = 99.97%, s = 0.02% (5 ampoules in duplicate, May 2010)
	Detector:	PDA at Max plot
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.1%, s = 0.4% (5 sub samples in duplicate, May 2010)
	Re-analysis:	Mean = 96.1%, s = 0.7% (5 sub samples in duplicate, May 2015)
	Re-analysis:	Mean = 97.8%, s = 0.3% (5 sub samples in duplicate, May 2018)
HPLC:	Column:	X-Bridge C-18, 5 µm (4.6 mm × 150 mm)
	Mobile Phase:	Acetonitrile/water (15:85)
	Flow Rate:	1.0 mL/min
	Detector:	PDA at 218 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.2%, s = 0.2% (10 sub samples in duplicate, December 2001)
	Re-analysis:	Mean = 99.4%, s = 0.1% (5 sub samples in duplicate, February 2007)
Karl Fischer analysis:	Moisture content 0.2% mass fraction (February 2010)	
	Moisture content < 0.1% mass fraction (May 2015)	
	Moisture content < 0.1% mass fraction (May 2018)	
	Moisture content 0.2% mass fraction (March 2021)	
	Moisture content < 0.1% mass fraction (July 2025)	
Thermogravimetric analysis:	Volatile content < 0.1% and non-volatile residue = 0.4% mass fraction (December 2001 and February 2007)	
³¹ P QNMR:	Instrument:	Bruker Avance III - 400
	Field strength:	161 MHz
	Solvent:	DMSO-d ₆ (2.50 ppm)
	Internal standard:	Triphenylphosphine oxide (100% m/m)
	Initial analysis:	Mean (42.3 ppm) = 98.3%, s = 1.5% (5 sub samples, May 2010)

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Finnigan MAT TSQ 700 with electrospray interface
	Operation:	Negative ion mode and positive ion mode, direct infusion at 5 μ L/min
	Ionisation:	Ammonium acetate buffer (7.5 mM, pH 7.5) / methanol (1:1)
	Ionisation:	ESI spray voltage at 3.2 kV for negative ion mode and 4.5 kV for positive ion mode.
	Peak:	318 [M-H] ⁺ , 304, 141 (100) <i>m/z</i> from negative ion mode
		337 [M+NH ₄] ⁺ , 320 ([M+H] ⁺ , 100), 304 <i>m/z</i> from positive ion mode
HRMS:		Found <i>m/z</i> 320.0384 [M-H] ⁺ C ₈ H ₁₉ NO ₆ PS ₂ ; requires <i>m/z</i> 320.0391 (2.2 ppm)
IR:	Instrument:	FT-IR, Biorad FTS3000MX
	Range:	4000-400 cm ⁻¹ , KBr powder
	Peaks:	3333, 3113, 2962, 2925, 1669, 1578, 1298, 1247, 1126, 1040, 770 cm ⁻¹ The IR spectrum shows a very strong absorbance at 1298 and 1126 cm ⁻¹ due to sulfone stretching.
¹ H NMR:	Instrument:	Bruker DMX-300
	Field strength:	300 MHz
	Solvent:	Benzene-d ₆
	Spectral data:	δ 1.51 (3H, d, <i>J</i> = 7.2 Hz), 2.60 (3H, d, <i>J</i> = 4.5 Hz), 3.10-3.20 (2H, m), 3.42 (3H, d, <i>J</i> _{PH} = 2.3 Hz), 3.46 (3H, d, <i>J</i> _{PH} = 2.3 Hz), 3.48-3.76 (2H, m), 3.59 (1H, q, <i>J</i> = 7.2 Hz), 6.31 (1H, s) ppm Ethyl acetate estimated at 0.04% mass fraction was observed in the ¹ H NMR Due to the phosphorus-hydrogen coupling the two phosphorothioate methyl ester resonances appear as two separate doublets.
¹³ C NMR:	Instrument:	Bruker DMX-300
	Field strength:	75.5 MHz
	Solvent:	Benzene-d ₆
	Spectral data:	δ 12.0, 23.3 (d, <i>J</i> _{CSP} = 3.8 Hz), 26.9, 51.7, 54.0 (d, <i>J</i> _{COP} = 6.0 Hz), 65.1, 166.0 ppm The signals of methoxy group and methylene group in the ¹³ C NMR spectrum are split due to phosphorus-carbon coupling.
Microanalysis:	Found:	C = 30.4%; H = 5.6%; N = 4.5%; S = 20.3% (September 2001)
	Calculated:	C = 30.1%; H = 5.7%; N = 4.4%; S = 20.1% (Calculated for C ₈ H ₁₈ NO ₆ PS ₂)