



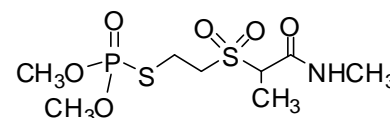
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1730: Vamidothion sulfone

Report ID: P1730.2021.02 (Bottled 160413)

Chemical Formula: C₈H₁₈NO₆PS₂

Molecular Weight: 319.3 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
01-AV-05	70898-34-9	99.6 ± 1.4%

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-propanyl]sulfonyl]ethyl) phosphorothioate.

Expiration of certification: The property values are valid till 10 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 solid 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 °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.
13 October 2022

This report supersedes any issued prior to 13 October 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 HPLC with CAD detection, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR spectroscopy. The purity value is calculated as per Equation 1.

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

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)
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)
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: Operation: Ionisation: Ionisation: Peak:	Finnigan MAT TSQ 700 with electrospray interface Negative ion mode and positive ion mode, direct infusion at 5 μ L/min Ammonium acetate buffer (7.5 mM, pH 7.5) / methanol (1:1) ESI spray voltage at 3.2 kV for negative ion mode and 4.5 kV for positive ion mode. 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: Range: Peaks:	FT-IR, Biorad FTS3000MX 4000-400 cm ⁻¹ , KBr powder 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: Field strength: Solvent: Spectral data:	Bruker DMX-300 300 MHz Benzene-d ₆ 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: Field strength: Solvent: Spectral data:	Bruker DMX-300 75.5 MHz Benzene-d ₆ 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: Calculated:	C = 30.4%; H = 5.6%; N = 4.5%; S = 20.3% (September 2001) C = 30.1%; H = 5.7%; N = 4.4%; S = 20.1% (Calculated for C ₈ H ₁₈ NO ₆ PS ₂)