Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

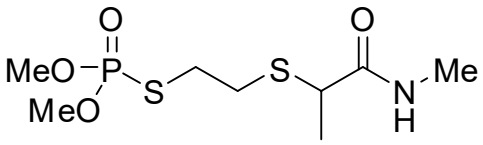
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1728: Vamidothion

Report ID: P1728.2024.01

Chemical Formula: $C_8H_{18}NO_4PS_2$

Molecular Weight: 287.3 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
01-AV-14	2275-23-2	97.9 ± 3.8%

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]sulfanyl}ethyl) phosphorothioate**.

Expiration of certification: The property values are valid till 25 July 2029, 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 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 all impurities are quantified as a mass fraction and subtracted from 100%.

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.
5 August 2024

This report supersedes any issued prior to 5 August 2024.

NATA Accreditation No. 198 / Corporate Site No. 14214.

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 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.

Supporting evidence is provided by elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 10 °C/min to 220 °C, 30 °C/min to 300 °C (5 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 98.1%, s = 0.2% (5 sub samples in duplicate, July 2008)
	Re-analysis:	Mean = 97.7%, s = 0.2% (5 sub samples in duplicate, April 2016)
	Re-analysis:	Mean = 97.3%, s = 0.1% (5 sub samples in duplicate, April 2019)
GC-FID:	Instrument:	Agilent 7890
	Column:	HP-35, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	100 °C (1 min), 20 °C/min to 220 °C (10 min), 30 °C/min to 300 °C (5 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 98.9%, s = 0.08% (5 sub samples in duplicate, July 2024)
	HPLC:	Instrument: Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Phenomenex C-18, 5 μm (2 mm × 150 mm)
	Mobile Phase:	12.5% Acetonitrile/87.5% of 10% Acetonitrile in MilliQ water
	Flow Rate:	0.2 mL/min
	Detector:	UV at 208 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.2%, s = 0.05% (10 sub samples in duplicate, January 2002)
	Re-analysis:	Mean = 98.4%, s = 0.06% (5 sub samples in duplicate, July 2008)
	Detector:	UV at Max Plot
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.3%, s = 0.05% (5 sub samples in duplicate, January 2008)
Thermogravimetric analysis:		Volatile content 1% and non-volatile residue < 0.4% mass fraction (December 2001)
Karl Fischer analysis:		Moisture content 0.4% mass fraction (July 2008) Moisture content 0.5% mass fraction (February 2016) Moisture content 1.2% mass fraction (April 2019) Moisture content 1.9% mass fraction (March 2020) Moisture content 0.8% mass fraction (April 2024)

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Finnigan MAT TSQ 700 with electrospray interface
	Operation:	Positive ion mode, direct infusion at 5 μL/min
	Solvent:	Ammonium acetate buffer (7.5 mM, pH 7.5) / methanol, 1:1
	Ionisation:	ESI spray voltage 4.5 kV (positive ion mode)
	Peak:	288 (MH^+) *m/z* (positive ion mode)
HRMS:		Found *m/z* 287.0423 (MH+) Calc *m/z* 287.0415 for $C_8H_{18}NO_4PS_2$
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400 cm^{-1}
	Peaks:	3310, 3084, 2953, 1655, 1558, 1250, 1021, 774 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-300
	Field strength:	300 MHz
	Solvent:	Benzene-d_6
	Key spectral data:	δ 1.39 (3H, d), 2.64 (3H, d), 3.23 (1H, q), 3.27 (3H, d, J_{PH} = 7.5 Hz), 3.31 (3H, d, J_{PH} =7.9 Hz) ppm
	Residual chloroform estimated at 0.15% mass fraction was observed in the ^{1}H NMR spectrum (July 2024).	
^{13}C NMR:	Instrument:	Bruker DMX-300
	Field strength:	75.5 MHz
	Solvent:	Benzene-d_6
	Spectral data:	δ 18.6, 26.9, 31.5 (d), 33.7 (d), 43.4, 54.1 (d), 172.7 ppm
Microanalysis:	Found:	C = 33.0%, H = 6.3%, N = 4.8%, S = 22.0% (September 2001)
	Calculated:	C = 33.4%, H = 6.3%, N = 4.9%, S = 22.3% (Calculated for $C_8H_{18}NO_4PS_2$)