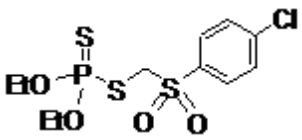
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA P1727: Carbophenothion sulfone

Report ID: P1727.2013.04

Chemical Formula: $C_{11}H_{16}ClO_4PS_3$

Molecular Weight: 374.9 g/mol



Property value

Batch No.	CAS No.	Purity estimate
01-AV-03	16662-85-4	99.6%

IUPAC name: S-{[(4-Chlorophenyl)sulfonyl]methyl} O,O-diethyl phosphorodithioate

Expiration of certification: The property values are valid till 20 May 2018, 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: Waxy 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 reference material should be used for qualitative analysis only.

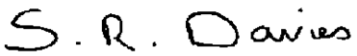
Instructions for use: Equilibrate the bottled material to room temperature before opening.

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: This material has demonstrated stability over a minimum period of five years. 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.



Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
14 September 2022

This report supersedes any issued prior to 14 September 2022.

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 purity value was obtained by mass balance from a combination of traditional analytical techniques, including HPLC with UV detection, Karl Fischer analysis and ^{1}H NMR spectroscopy. The purity value is calculated as per Equation 1

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

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

Supporting evidence is provided by elemental microanalysis.HPLC: Instrument: Waters alliance 2650

	Column:	Phenomenex C-18 (2), 5 µm (150 mm × 2 mm)
	Column oven:	55 °C
	Mobile Phase:	Acetonitrile/MilliQ water (67:33 v/v) Flow rate: 0.3 mL/min
	Detector:	Waters 2998 PDA operating at 231 nm
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.04% (10 sub samples in duplicate, November 2001)
	Re-analysis:	Mean = 99.9%, s = 0.02% (5 sub samples in duplicate, July 2008)
HPLC:	Instrument:	Waters alliance 2650
	Column:	Phenomenex C-18 (2), 5 µm (150 mm × 2 mm)
	Column oven:	55 °C
	Mobile Phase:	Acetonitrile/MilliQ water (60:40 v/v) Flow rate: 0.3 mL/min
	Detector:	Waters 2998 PDA operating at 231 nm
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.9 %, s = 0.01% (5 sub samples in duplicate, May 2013)
Karl Fischer analysis:		Moisture content is 0.3% mass fraction (July 2008) Moisture content is < 0.1 % mass fraction (May 2013)
Thermogravimetric analysis:		Volatile content < 0.5% mass fraction (December 2001)

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 3 µL/min
	Ionisation:	ESI spray voltage at 3.5 kV for -ve ion mode and 4.5 kV for +ve ion mode
	Peak:	373 (M^-), 175 (100) *m/z* from negative ion mode 392 (MNH_4^+), 375 (MH^+, 100) *m/z* from positive ion mode
IR:	Instrument:	FT-IR, Biorad Merlin
	Range:	4000-400 cm^{-1}, as film
	Peaks:	2960, 2900, 1581, 1474, 1395, 1320, 1151, 1005, 970 cm^{-1}
^1H NMR:	Instrument:	Bruker DMX-300
	Field strength:	300 MHz
	Solvent:	d_6-Benzene (7.27 ppm)
	Spectral data:	δ 1.065, 1.067 (6H, t), 3.87, 3.99 (2H, m), 4.00-4.13 (2H, m), 4.19, 4.25 (2H, d), 6.94 (2H, d), 7.64 (2H, d) ppm
^{13}C NMR:	Instrument:	Bruker DMX-300
	Field strength:	75.5 MHz
	Solvent:	d_6-Benzene (128.4 ppm)
	Spectral data:	δ 16.0 (d), 57.7 (d), 65.2 (d), 129.8, 131.1, 137.2, 141.0 ppm
Microanalysis:	Found:	C = 35.5%; H = 4.5%; S = 25.6% (September 2001)
	Calculated:	C = 35.2%; H = 4.3%; S = 25.7% (Calculated for $C_{11}H_{16}ClO_4PS_3$)