



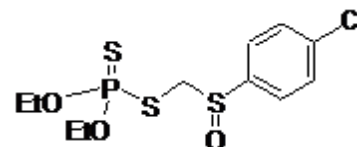
# REFERENCE MATERIAL PRODUCT INFORMATION SHEET

## NMIA P1726: Carbophenothion sulfoxide

Report ID: P1726.2013.04

Chemical Formula: C<sub>11</sub>H<sub>16</sub>ClO<sub>3</sub>PS<sub>3</sub>

Molecular Weight: 358.9 g/mol



### Property value

| Batch No. | CAS No.    | Purity estimate |
|-----------|------------|-----------------|
| 01-AV-02  | 17297-40-4 | 99.2%           |

**IUPAC name:** S-[(4-Chlorophenyl)sulfinyl]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:** Off-white 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 °C in a closed container in a dry, dark area.

**Stability:** This material has demonstrated stability over a minimum period of five years. The material is stable in the form provided if stored as recommended.

It is prone to oxidation when taken into solution. Standard solutions prepared from this material should be stored out of direct light at 4 °C and monitored regularly for oxidation to carbophenothion sulfone.

**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.  
14 September 2022

This report supersedes any issued prior to 14 September 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.

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### 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 <sup>1</sup>H NMR. The purity value is calculated as per Equation 1.

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

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

Supporting evidence is provided by elemental microanalysis.

|                             |  |   |                       |
|-----------------------------|--|---|-----------------------|
| HPLC                        | Column:  | Phenomenex C-18 (2), 5 µm (150 mm × 2 mm)                           |                       |
|                             | Column oven:                                       | Ambient   |                       |
|                             | Mobile Phase:                                      | Acetonitrile/MilliQ water (60:40 v/v)                               | Flow rate: 0.3 mL/min |
|                             | Detector:  | UV at 250 nm  |                       |
|                             | Relative peak area of the main component:          |   |                       |
|                             | Initial analysis:                                  | Mean = 99.4%, s = 0.12% (10 sub samples in duplicate, January 2001) |                       |
|                             | Re-analysis:                                       | Mean = 99.8%, s = 0.065% (5 sub samples in duplicate, July 2008)    |                       |
|                             | Re-analysis:                                       | Mean = 99.6 %, s = 0.05% (5 sub samples in duplicate, May 2013)     |                       |
| Karl Fischer analysis:      | Moisture content is 0.5% mass fraction (July 2008) |   |                       |
|                             | Moisture content is 0.1% mass fraction (May 2013)  |   |                       |
| Thermogravimetric analysis: | Volatiles content < 0.4% (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 $\mu$ L/min  |
|                      | Solvent:        | ammonium acetate buffer (7.5 mM, pH 7.5) / methanol, 1:1   |
|                      | Ionisation:     | ESI spray voltage at 3.5 kV for -ve ion mode and 4.5 kV for +ve ion mode.  |
|                      | Peaks:          | 417 (MCH <sub>3</sub> COO <sup>-</sup> ), 357 (M <sup>-</sup> ), 329 (M-Et, 100), 159 m/z. (-ve ion mode)<br>376 (M+NH <sub>4</sub> <sup>+</sup> ), 359 (M+H <sup>+</sup> , 100) m/z. (+ve ion mode) |
| IR:                  | Instrument:     | FT-IR, Biorad Merlin   |
|                      | Range:          | 4000-400 cm <sup>-1</sup> , as film  |
|                      | Peaks:          | 2983, 2902, 1575, 1475, 1391, 1296, 1161, 1090, 1056, 1009, 966 cm <sup>-1</sup>   |
| <sup>1</sup> H NMR:  | Instrument:     | Bruker DMX-300   |
|                      | Field strength: | 300 MHz  |
|                      | Solvent:        | benzene- <i>d</i> <sub>6</sub> (7.27 ppm)  |
|                      | Spectral data:  | $\delta$ 1.09, 1.12 (6H, t), 3.73, 3.80 (1H, d), 4.00-4.25 (5H, m), 7.06 (2H, d), 7.35 (2H, d) ppm   |
| <sup>13</sup> C NMR: | Instrument:     | Bruker Avance III-500  |
|                      | Field strength: | 126 MHz  |
|                      | Solvent:        | benzene- <i>d</i> <sub>6</sub> (128.4 ppm)   |
|                      | Spectral data:  | $\delta$ 16.1 (d), 59.5 (d), 65.0 (d), 65.1 (d), 126.5, 129.8, 137.8, 142.9 ppm  |
| Microanalysis:       | Found:          | C = 37.0%, H = 4.4%, S = 27.0% (September 2001)  |
|                      | Calculated:     | C = 36.8%, H = 4.5%, S = 26.8% (Calculated for C <sub>11</sub> H <sub>16</sub> ClO <sub>3</sub> PS <sub>3</sub> )  |