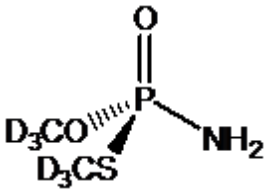
# DEUTERATED INTERNAL STANDARD PRODUCT INFORMATION SHEET

## NMIA P1712: d6-Methamidophos

Report ID: P1712.2018.04

Chemical Formula: $C_2H_2D_6NO_2PS$

Molecular Weight: 147.2 g/mol




## Property value

| Batch No. | CAS No. | Purity estimate |
|---|---|---|
| 01-AV-01 | Not available | 96.7% |

**IUPAC name:** $d_6$-O, S-Dimethyl phosphoramidothioate

**Expiration of certification:** The property values are valid till 28 June 2023, 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 powder prepared by synthesis, 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:** The isotopic purity of this material is an estimate only. This material should be considered for use as an internal standard 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:** 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 GC-FID 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.

## Characterisation Report:

Purity estimate obtained from quantitative NMR using a certified internal standard of triphenylphosphine oxide to quantify the amount of $d_6$-methamidophos in the sample. Supporting evidence is provided by elemental microanalysis. Impurity estimates by GC-FID and thermogravimetric analysis are also provided.

| | | |
|---|---|---|
| Isotopic Purity: | $d_4 \approx 99.4\%$ | $[= 100 \times d_6/(d_0 + d_1 + d_2 + d_3 + d_4 + d_5 + d_6)]$ |
| | $d_0 \approx 0\%$ | $[= 100 \times (d_0/d_0 + d_1 + d_2 + d_3 + d_4 + d_5 + d_6)]$ |

| | | |
|---|---|---|
| GC-FID: | Instrument: | HP5890 |
| | Column: | BP-20 Capillary, 25 m × 0.33 mm I.D. × 0.5 μm |
| | Program: | 130 °C (1 min), 15 °C/min to 220 °C (4 min) |
| | Injector: | 180 °C |
| | Detector Temp: | 250 °C |
| | Carrier: | Helium |
| | Split ratio: | 20/1 |
| | Relative peak area of the main component: | |
| | Initial analysis: | Mean = 100.0%, s = 0.00% (10 sub samples in duplicate, October 2001) |
| Thermogravimetric analysis: | | Moisture content ≤ 0.3% total. Inorganic residue was impossible to measure due to incomplete combustion. |
| $^{31}$P QNMR: | Instrument: | Bruker DMX-500 |
| | Field strength: | 202 MHz |
| | Solvent: | $CDCl_3$ |
| | Internal standard: | Triphenylphosphine oxide (100.0% mass fraction) |
| | Initial analysis: | Mean (37.1 ppm) = 96.0%, s = 1.1% (4 sub samples, June 2002) |
| | Re-analysis: | Mean (37.1 ppm) = 97.2%, s = 1.2% (5 sub samples, April 2010) |

## Spectroscopic and other characterisation data

| | | | |
|---|---|---|---|
| GC-MS: | Instrument: | HP 6890/5973 | |
| | Column: | Zebron ZB-5, 30 m × 0.25 mm I.D. × 0.25 μm | |
| | Program: | 80°C, 10 °C/min to 250°C | |
| | Injector: | 180 °C (1 min) | Transfer line temp: 280 °C |
| | Carrier: | Helium | Split ratio: 20/1 |
| | The retention time of the parent compound is reported along with the major peaks in the mass spectrum. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak. | | |
| | Parent (6.4min): | 147 ($M^+$, 40), 129 (3), 115 (5), 99 (45), 97 (100), 83 (7), 65 (15), 51 (18) *m/z* | |
| | Deuteration yield (by SIM analysis of the parent compound, mean of 7 replicates) | | |
| | Column: | Zebron ZB-5, 30 m × 0.25 mm I.D. × 0.30 μm | |
| | Program: | 80°C, 10 °C/min to 250 °C | |
| | (6.0 min): | (Deuteration state, % rel. to $d_6$-methamidophos at 147 *m/z*) 141 ($d_0$, 0), 142 ($d_1$, 0), 143 ($d_2$, 0), 144 ($d_3$, 0.01), 145 ($d_4$, 0.06), 146 ($d_5$, 0.44), 147 ($d_6$, 99.4) *m/z* | |
| TLC: | Conditions: | Kieselgel 60$F_{254}$. Ethyl acetate/acetone (3/1)<br>Single spot observed, $R_f$ = 0.2 | |
| IR: | Instrument: | FT-IR, Biorad WIN FTS40 | |
| | Range: | 4000-400 $cm^{-1}$, KBr pellet | |
| | Peaks: | 2258, 2200, 2134, 2078, 1561, 1226, 1068, 1056, 936, 738 $cm^{-1}$ | |
| $^1$H NMR: | Instrument: | Bruker Avance III-500 | |
| | Field strength: | 500 MHz | |
| | Solvent: | $CDCl_3$ (7.26 ppm) | |
| | Spectral data: | δ 3.4 (2H, s, $NH_2$) ppm | |
| $^2$H NMR: | Instrument: | Bruker Avance III-500 | |
| | Field strength: | 77 MHz | |
| | Solvent: | $CDCl_3$ | |
| | Spectral data: | δ 2.27 (3D, d, $J_{DP}$ = 14.6 Hz), 3.74 (3D, d, $J_{DP}$ = 12.6 Hz) ppm | |
| $^{31}$P NMR: | Instrument: | Bruker Avance III-500 | |
| | Field strength: | 121.5 MHz | |
| | Solvent: | $CDCl_3$ | |
| | Spectral data: | δ 37.1 (1P, s) ppm | |
| Melting point: | | 43-44 °C | |
| Microanalysis: | Found: | C = 16.5%; H/D = 9.6%; N = 9.4% (November 2001) | |
| | Calculated: | C = 16.3%; H/D = 9.6%; N = 9.5% (Calculated for $C_2H_2D_6NO_2PS$) | |