Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

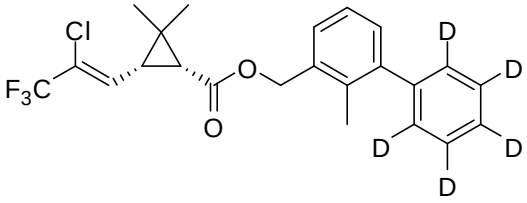
DEUTERATED INTERNAL STANDARD PRODUCT INFORMATION SHEET

NMIA P1806: d5-Bifenthrin

Report ID: P1806.2023.01 (Ampouled 231130)

Chemical Formula: $C_{23}H_{17}D_5ClF_3O_2$

Molecular Weight: 427.9 g/mol



Property value

Batch No.	CAS No.	Mass per ampoule
11-AV-03	**Not available**	**969 ± 27 µg**

Synonym: d_5-2-Methylbiphenyl-3-ylmethyl (*Z*)-(1*RS*)-*cis*-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate.

Expiration of certification: The property values are valid till 5 December 2026, i.e. three 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 shelf life does not apply to ampoules that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials.

Description: The compound is supplied as a dried aliquot in a sealed ampoule under an atmosphere of argon. The deuterated internal standard is intended for a single use to prepare a standard solution containing P1806. The material was prepared by synthesis and certified for identity and purity by NMIA. The main component of this material is d_5-bifenthrin. d_4-, d_3-, d_2-, d_1- and d_0-bifenthrin are also present. The stated mass of the analyte per ampoule represents the approximate combined masses of deuterated (d_5, d_4, d_3, d_2 and d_1) and d_0-bifenthrin in the material.

Intended use: The isotopic purity of this material is an estimate only. This material should be considered for use as an internal standard only and is not intended for use as a calibrator. The material does not have certified reference material status as metrological traceability of the stated purity value to the SI unit for mass (kg) has not been established.

Instructions for use: Open the ampoule and carefully rinse the interior at least three times with a suitable organic solvent (e.g. methanol). This will transfer approximately 969 ± 27 µg of anhydrous bifenthrin (d_5, d_4, d_3, d_2, d_1 and d_0). The mass of analyte in each ampoule is calculated from the assigned purity of the bulk and the concentration of bulk material in a stock solution used to prepare the ampoules.

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: In the absence of long term stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated from stability trials conducted on similar materials by NMI Australia over the last ten 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 GC-FID on seven randomly selected ampoules 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 December 2023

This report supersedes any issued prior to14 December 2023.

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

Characterisation Report:

GC-FID:	Instrument:	Varian CP-3800
	Column:	DB-17, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	150 °C (1 min), 30 °C/min to 250 °C (10 min), 30 °C/min to 280 °C (5 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component	
	Initial analysis:	Mean = 96.9%, s = 0.05% (7 ampoules in duplicate, December 2023)

The following analytical data was obtained on the bulk material subsequently used in the preparation of the ampoules.

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 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 qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

The main component of this material is d_5-bifenthrin. d_4-, d_3-, d_2-, d_1- and d_0- bifenthrin are also present. The stated chemical purity of the analyte represents the combined mass fractions of deuterated (d_5, d_4, d_3, d_2 and d_1) and d_0-bifenthrin in the material.

The isotopic purity of this material is an estimate only. This material should be considered for use as an internal standard only.

Isotopic Purity:	$d_5 \approx 94$ % [= $d_4/(d_4 + d_3 + d_2 + d_1 + d_0)$ x 100]	
	$d_0 < 0.1$ % [= $d_0/(d_4 + d_3 + d_2 + d_1 + d_0)$ x 100]	
GC-FID:	Instrument:	Varian CP-3800
	Column:	TG-17MS, 29.9 m × 0.32 mm I.D. × 0.25 µm
	Program:	150 °C (1 min), 30 °C/min to 250 °C (10 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 95.7%, s = 0.03% (10 sub samples in duplicate, June 2011)
	Re-analysis:	Mean = 95.1%, s = 0.11% (5 sub samples in duplicate, May 2012)
	Re-analysis:	Mean = 95.2%, s = 0.3% (5 sub samples in duplicate, April 2015)
GC-FID:	Instrument:	Varian CP-3800
	Column:	DB-17, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	150 °C (1 min), 30 °C/min to 250 °C (10 min), 30 °C/min to 280 °C (5 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component	
	Initial analysis:	Mean = 95.8%, s = 0.3% (5 sub samples in duplicate, March 2020)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (June 2011, May 2012 and March 2015)
Thermogravimetric analysis:		Non volatile residue < 0.2% mass fraction (June 2011). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material.

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 µm
	Program:	180 °C (1 min), 10 °C/min to 300 °C (2 min)
	Injector:	250 °C
	Split ratio:	30/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention time of the parent compound isreported with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (9.8 min):	187 (18), 186 (100), 185 (9), 172 (6), 171 (21), 170 (19), 169 (14) *m/z*
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 µm
	Program:	50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min)
	Injector:	150 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Split ratio:	50/1
	Solvents detected:	Pentane
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/acetone (9:1) Single spot observed, R_f = 0.50 Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powde
	Peaks:	3093, 3004, 2970, 2290, 2268, 1719, 1654, 1470, 1411, 1382, 1357, 1296, 1274, 1198, 1149, 1083, 952, 889, 727, 554 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 1.31 (3H, s), 1.32 (3H, s), 2.07 (1H, d, *J* = 8.4 Hz), 2.19 (1H, t, *J* = 8.9 Hz), 2.23 (3H, s), 5.19 (1H, d, *J* = 12.6 Hz), 5.23 (1H, d, *J* = 12.6 Hz), 6.97 (1H, s, *J* = 9.3 Hz), 7.24-7.28 (2H, m), 7.35 (1H, dd, *J* = 2.0, 6.7 Hz) ppm
	n-Pentane at 0.03 % mass fraction was determined from the ^{1}H NMR spectrum.	
^{13}C NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	151 MHz
	Solvent:	$CDCl_3$ (77.0 ppm)
	Spectral data:	δ 15.0, 16.2, 28.4, 28.7, 30.9, 32.9, 65.4, 120.4 (quartet, *J* = 271 Hz), 121.8 (quartet, *J* = 37 Hz), 125.6, 126.4 (t, *J* = 24 Hz), 127.6 (t, *J* = 24 Hz), 128.4, 128.9 (t, *J* = 24 Hz), 130.0 (quartet, *J* = 4 Hz), 130.4, 134.2, 134.4, 141.6, 143.0, 170.1 ppm
Microanalysis:	Found:	C = 64.5 %; H/D = 5.3 % (June, 2011)
	Calculated:	C = 64.6 %; H/D = 5.3 %; (Calculated for $C_{23}H_{17}D_5ClF_3O_2$)