Australian Government
Department of Industry, Science and Resources

National Measurement Institute

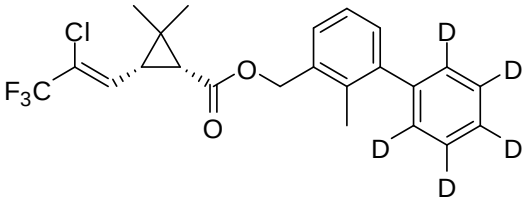
DEUTERATED INTERNAL STANDARD PRODUCT INFORMATION SHEET

NMIA P1806: d5-Bifenthrin

Report ID: P1806.2020.03

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

Molecular Weight: 427.9 g/mol



Property value

Batch No.	CAS No.	Purity by GC-FID
11-AV-03	**Not available**	**95.8 ± 1.8%**

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 26 March 2025, 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 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 five 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.
20 October 2022

This report supersedes any issued prior to 18 October 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 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 $C_{23}H_{17}D_5ClF_3O_2$)