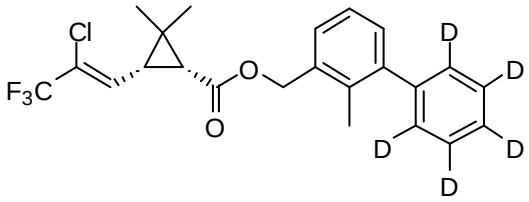
Australian Government

National Measurement Institute

REFERENCE MATERIAL ANALYSIS REPORT

Report ID: P1806.2015.01

Compound Name: **d_5-Bifenthrin**
Collection number: P1806
Chemical Formula: $C_{23}H_{17}D_5ClF_3O_2$
CAS Number: NA
Structure:



Description: Off white solid
Batch number: 11-AV-03
Molecular Weight: 427.9
Batch production completed: June 2011

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

Purity (mass fraction): 95.1 ± 1.4% (95 % coverage interval)

Purity estimate obtained from traditional analytical techniques. The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by GC-FID, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR. Supporting evidence is provided by headspace GC-MS analysis of occluded solvent 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: $d_5 \approx 94\ \%$ [$= d_4/(d_4 + d_3 + d_2 + d_1 + d_0) \times 100$]
$d_0 < 0.1\ \%$ [$= d_0/(d_4 + d_3 + d_2 + d_1 + d_0) \times 100$]
[from SIM analysis of the d_5-2-methylbiphenyl-3-yl-methanol precursor]

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 response of 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)

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.

Karl Fischer analysis: Moisture content < 0.1% mass fraction (June 2011, May 2012 and March 2015)

Spectroscopic and other characterisation data

GC-MS:	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	Transfer line temp: 280 °C
	Carrier:	Helium, 1.0 mL/min	Split ratio: 30/1

The retention time of the parent compound is reported along with the major peaks in the mass spectrum. The latter are reported as mass to 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
The parent compound co-elutes with a comparison sample of bifenthrin.

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 powder
	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) Calc: C = 64.6 %; H/D = 5.3 %; (Calculated for $C_{23}H_{17}D_5ClF_3O_2$)

Expiration of certification
The property values are valid till 30th April 2020, 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.

The long-term stability of the compound in solution has not been examined.

This material has demonstrated stability over a minimum period of five years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from annual stability trials.

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 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.

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.

Intended Use
For ***in vitro*** laboratory analysis only.

Caution
Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice
Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 5 May 2015

Characterisation data and property values specified in this report supersede those in all reports issued prior to 5th May 2015.