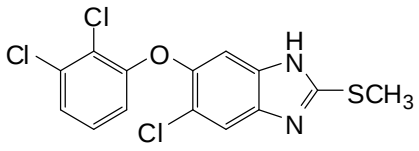
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA P1788: Triclabendazole

Report ID: P1788.2011.05

Chemical Formula: $C_{14}H_9Cl_3N_2OS$

Molecular Weight: 359.7 g/mol



Property value

Batch No.	CAS No.	Purity estimate
04-AV-01	68786-66-3	98.5 ± 1.10%

IUPAC name: 6-Chloro-5-(2,3-dichlorophenoxy)-2-(methylthio)-1H-benzimidazole.

Expiration of certification: The property values are valid till 1 February 2014, 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 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 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 ºC in a closed container in a dry, dark area.

Stability: This material has demonstrated stability over a minimum period of three 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 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.
13 October 2022

This report supersedes any issued prior to 13 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. Impurities of related structure were assessed by HPLC with UV detection at 305 nm. The purity value was obtained by mass balance from a combination of traditional analytical techniques, including HPLC with UV detection, 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 elemental microanalysis.

HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	Ascentis C-18, 2.7 μm (4.6 mm x 150 mm)
	Column oven:	Ambient
	Mobile Phase:	Acetonitrile/MilliQ water (65:35 v/v) The aqueous phase was buffered at pH 4.2 using 20mM NH_4OAc and AcOH
	Flow rate:	0.3 mL/min
	Detector:	Shimadzu SPD-M20A PDA operating at 305 nm
	Relative peak area of the main component:	
	Initial analysis:	Mean = 98.6%, s = 0.04% (10 sub samples in duplicate, February 2011)
HPLC:	Instrument:	Waters PDA 996
	Column:	Phenomenex C18, 5 μm (2 mm x 150 mm)
	Column oven:	Ambient
	Mobile Phase:	Acetonitrile/Milli-Q water (53:47 v/v) The aqueous phase was buffered at pH 4.2 using 20mM NH_4OAc and AcOH
	Flow rate:	0.3 mL/min
	Detector:	Waters 2998 PDA operating at 225 nm
	Relative peak area of the main component:	
	Initial analysis:	Mean = 94.9%, s = 0.3% (10 sub samples in duplicate, May 2004)
	Re-analysis:	Mean = 95.1%, s = 0.02% (5 sub samples in duplicate, October 2007)
Karl Fischer analysis:		Moisture content 0.1% mass fraction (October 2007, December 2010)
Thermogravimetric analysis:		Volatile content < 0.1%. The non-volatile content was not determined due to the nature of the material (June 2002 & November 2006).

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 5 µL/min
	Ionisation:	ESI spray voltage at 3.2 kV positive ion
	Desolvation temp:	200 ºC
	EM voltage:	650 V
	Cone voltage:	35 V
	Peak:	363 (33), 361 (100), 359 (97), 105 (52), 102 (48), 99 (30) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Acetone/ethyl acetate (1:1) Single spot observed, R_f = 0.6. Visualisation with 254 nm.
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1}, KBr pellet.
	Peaks:	3105, 1626, 1576, 1450, 1422, 1382, 1334, 1257, 1153, 1022, 915, 854, 803, 769, 697, 662 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Gyro-300
	Field strength:	300 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 2.69 (3H, s), 6.66 (1H, dd, *J* = 1.1, 8.3 Hz), 7.25 (1H, t, *J* = 8.3 Hz), 7.31 (1H, s), 7.35 (1H, dd, *J* = 1.5, 8.3 Hz), 7.68 (1H, s) ppm
^{13}C NMR:	Instrument:	Bruker Gyro-300
	Field strength:	75 MHz
	Solvent:	MeOH-d_4 (49 ppm)
	Spectral data:	δ 13.8, 115.2, 118.5, 120.9, 124.2, 128.6, 132.8, 144.7, 154.2, 154.6 ppm
Melting point:		173-175 °C
Microanalysis:	Found:	C = 46.7%; H = 2.6%; N = 7.9% (July, 2004)
	Calculated:	C = 46.8%; H = 2.5%; N = 7.8% (Calculated for $C_{14}H_9Cl_3N_2OS$)