Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

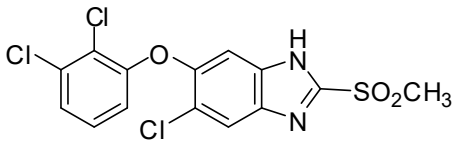
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1684: Triclabendazole sulfone

Report ID: P1684.2020.03

Chemical Formula: $C_{14}H_9Cl_3N_2O_3S$

Molecular Weight: 391.7 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
01-AV-07	106791-37-1	99.1 ± 0.5%

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit (k = 2).

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

Expiration of certification: The property values are valid till 27 October 2030, i.e. ten 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 material will be re-tested on an annual basis to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

Description: White crystalline solid 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 certified reference material is suitable for use as a primary calibrator.

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.

Metrological traceability: The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance approach all impurities are quantified as a mass fraction and subtracted from 100%.

Stability: This material has demonstrated stability over a minimum period of 5 years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials. 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.
12 September 2022

This report supersedes any issued prior to 12 September 2022.

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

Legal notice: Terms and Conditions associated with the provision of this reference material can be found on the NMIA website.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified 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:	Waters alliance 2695
	Column:	Alltech C-18, 5 μm (4.6 mm × 150 mm)
	Column oven:	40°C
	Mobile Phase:	A = 20 mM NH_4OAc pH 4.2, B = Acetonitrile 0-8 min 55% B; 8-9 min 55-80% B; 9-14 min 80%B; 14-15 min 80-55%B.
	Flow rate:	1.0 mL/min
	Detector:	Waters 2998 PDA operating at 225 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.02% (5 sub samples in duplicate, October 2020)
HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Alltech C-18, 5 μm (4.6 mm × 150 mm)
	Column oven:	Ambient
	Mobile Phase:	60% Acetonitrile/40% 20 mM NH_4OAc pH 4.2
	Flow rate:	1.0 mL/min
	Detector:	Waters 2998 PDA operating at 225 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.6%, s = 0.05% (10 sub samples in duplicate, July 2001)
	Re-analysis:	Mean = 99.9%, s = 0.02% (5 sub samples in duplicate, February 2011)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, January 2016)
Karl Fischer analysis:		Moisture content < 0.2% mass fraction (February 2008, 2011, January 2016 and November 2020)
Thermogravimetric analysis:		Volatiles content and non-volatile residue < 0.3% mass fraction (December 2001)

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Finnigan MAT TSQ 700 with electrospray interface
	Operation:	Positive and negative ion mode, direct infusion at 5 μL/min
	Ionisation:	ESI spray voltage at 4.5 kV positive ion, 2.5 kV positive ion
	Peak:	389, 391, 393 *m/z* (main isotope peaks for $[M-H]^-$, negative ion mode) 391, 393, 395 *m/z* (main isotope peaks for $[M+H]^+$, positive ion mode)
HRMS:	Found:	389.9405
	Calculated:	389.9399 for $C_{14}H_9{}^{35}Cl_3N_2O_3S$
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400 cm^{-1}, KBr disc
	Peaks:	3209, 1576, 1447, 1428, 1334, 1250, 1196, 1141, 1026, 977, 914, 866, 772 cm^{-1}
^1H NMR:	Instrument:	Bruker DMX-300
	Field strength:	300 MHz
	Solvent:	DMSO-d_6 (2.54 ppm)
	Spectral data:	δ 3.30 (1H, br s), 3.51 (3H, s), 6.83 (1H, dd), 7.31 (1H, t), 7.44 (1H, dd), 7.50 (1H, s), 8.03 (1H, s) ppm
		^1H NMR indicates the presence of acetone and chloroform at 0.16% and 0.51% mass fraction respectively (August 2006 and November 2020)
^{13}C NMR:	Instrument:	Bruker DMX-300
	Field strength:	75.5 MHz
	Solvent:	DMSO-d_6 (39.9 ppm)
	Spectral data:	δ 42.4, 108.6 (br), 116.7, 118.3 (br), 122.0, 122.4, 125.5, 129.2, 133.4, 138.0 (br), 138.0 (br), 147.9, 151.5, 154.3 ppm
Melting point:		201-203 °C
Microanalysis:	Found:	C = 42.9%; H = 2.2%; N = 7.0% (June 2001)
	Calculated:	C = 42.9%; H = 2.3%; N = 7.2% (Calculated for $C_{14}H_9Cl_3N_2O_3S$)