Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

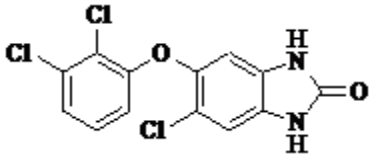
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1725b: Ketotriclabendazole

Report ID: P1725b.2021.02

Chemical Formula: $C_{13}H_7Cl_3N_2O_2$

Molecular Weight: 329.6 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
04-AV-02	Not Available	98.9 ± 0.6%

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)-1,3-dihydro-2H-benzimidazol-2-one

Expiration of certification: The property values are valid till 3 June 2031, 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.

Description: 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: 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 ten 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 seven 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.
14 September 2022

This report supersedes any issued prior to 14 September 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 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 qualitative elemental microanalysis.

HPLC:	Instrument:	Waters alliance 2695 or Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Alltima C-18, 5 μm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	Acetonitrile/MilliQ water A = MilliQ water; B = Acetonitrile 0-12 min 46% B, 12-14 min 46-75% B, 14-19 min 75% B, 19-20 min 75-46% B, 20-27 min 46% B The aqueous phase was buffered at pH 4.2 using 20 mM NH_4OAc and AcOH
	Flow rate:	1.0 mL/min
	Detector:	Waters 2998 PDA operating at 230 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.1%, s = 0.02% (7 sub samples in duplicate, September 2004)
	Re-analysis:	Mean = 98.9%, s = 0.08% (5 sub samples in duplicate, April 2008)
	Re-analysis:	Mean = 99.2%, s = 0.02% (5 sub samples in duplicate, May 2011)
	Re-analysis:	Mean = 99.2%, s = 0.05% (5 sub samples in duplicate, July 2016)
	Re-analysis:	Mean = 99.3%, s = 0.03% (5 sub samples in duplicate, June 2021)
Karl Fischer analysis:		Moisture content < 0.3% mass fraction (April 2008 and May 2011) Moisture content < 0.4% mass fraction (July 2016 and April 2021)
Thermogravimetric analysis:		Volatiles content < 0.1% and non-volatile residue < 0.2% mass fraction (September 2004 and April 2008)

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 μL/min
	Solvent	Ammonium acetate buffer (50 mM, pH ~4.2) / Methanol (1:1)
	Ionisation:	ESI spray voltage at 3.2 kV positive ion
	Desolvation temp:	200 °C
	Capillary voltage:	3.2 kV
	Multiplier:	650 V
	Peak:	329, 331, 333 *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Ethyl acetate/acetone (1:1) Single spot observed, R_f = 0.3. Visualisation with UV at 254 nm.
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1},KBr pellet
	Peaks:	3163, 3080, 2772, 1705, 1563, 1489, 1448, 1358, 1252, 1152, 1028, 878, 764, 602 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz
	Solvent:	DMSO-d_6 (2.5 ppm)
	Spectral data:	δ 6.68 (1H, d, *J* = 8.3 Hz), 6.80 (1H, s), 7.09 (1H, s), 7.25 (1H, dd, *J* = 7.9, 8.3 Hz), 7.35 (1H, d, *J* = 7.9 Hz) ppm
^{13}C NMR:	Instrument:	Bruker DMX-600
	Field strength:	150 MHz
	Solvent:	DMSO-d_6 (39.5 ppm)
	Spectral data:	δ 103.0, 109.9, 115.4, 116.8, 121.1, 124.5, 128.2, 128.9, 133.0, 144.1, 154.8, 155.6 ppm
Melting point:		295-297 °C
Microanalysis:	Found:	C = 47.0%; H = 2.3%; N = 8.4% (September, 2004)
	Calculated:	C = 47.4%; H = 2.1%; N = 8.5% (Calculated for $C_{13}H_7Cl_3N_2O_2$)