Australian Government
Department of Industry, Science and Resources

National Measurement Institute

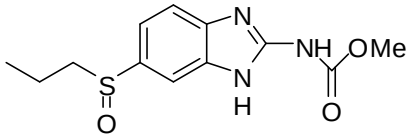
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA P1792: Albendazole Sulfoxide

Report ID: P1792.2020.03

Chemical Formula: $C_{12}H_{15}N_3O_3S$

Molecular Weight: 281.3 g/mol



Property value

Batch No.	CAS No.	Purity estimate
03-AV-07	54029-12-8	97.4 ± 1.8%

IUPAC name: Methyl [5-(propylsulfinyl)-1H-benzimidazol-2-yl]carbamate

Expiration of certification: The property values are valid till 24 June 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.

Description: Off-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 five years. It is potentially susceptible to oxidation to albendazole sulfone when taken into solution and exposed to air. Standard solutions prepared from this material should be stored out of direct light at 4 ˚C and monitored regularly for oxidation.

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.
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. The purity value was obtained by mass balance from a combination of traditional analytical techniques, including HPLC with UV detection, 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 Model 1525 Binary pump, 717 plus autosampler or Shimadzu Binary pump LC-20AB, SIL-20 A HT autosamplerThermo Scientific UltiMate 3000
	Column:	X-Bridge C-18, 5 μm (4.6 mm × 150 mm)
	Column oven:	40 °C
	Mobile Phase:	A= Milli-Q water with 0.05% TFA, B= Methanol with 0.05% TFA 0-15min 26% B, 15-16 min 26-80% B, 16-21 min 80% B, 21-22 min 80-26% B, 22-30 min 26% B
	Flow rate:	1.0 mL/min
	Detector:	Waters 2998 or Shimadzu SPD-M20A PDA operating at 226 nm
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.3%, s = 0.02% (7 sub samples in duplicate, July 2003)
	Re-analysis:	Mean = 99.5%, s = 0.03% (3 sub samples in duplicate, November 2004)
	Re-analysis:	Mean = 99.3%, s = 0.04% (5 sub samples in duplicate, July 2006)
	Re-analysis:	Mean = 99.5%, s = 0.01% (5 sub samples in duplicate, July 2007)
	Re-analysis:	Mean = 99.3%, s = 0.10% (5 sub samples in duplicated, July 2010)
	Re-analysis:	Mean = 99.5%, s = 0.02% (5 sub samples in duplicated, July 2015)
	Re-analysis:	Mean = 99.5%, s = 0.02% (5 sub samples in duplicated, June 2020)
	Karl Fischer analysis:	Moisture content 2.1% mass fraction (July 2006 & 2010) Moisture content 2.0% mass fraction (June 2015 and 2020)

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 2.8 kV positive ion
	Desolvation temp:	200 °C
	EM voltage:	650 V
	Peak:	304.1 ($[M+Na]^+$, 20), 282.0 ($[M+H]^+$, 100) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Methanol/chloroform (1:9) Single spot observed, R_f = 0.3. Visualization with UV at 254 nm.
IR:	Instrument:	BioRad FTS 3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3333, 2959, 1703, 1649, 1596, 1460, 1430, 1374, 1103 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX600
	Field strength:	600 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 0.93 (3H, t), 1.46 (1H, m), 1.58 (1H, m), 2.74 (1H, m), 2.82 (1H, m), 3.77 (3H, s), 7.31 (1H, dd), 7.55 (1H, d), 7.69 (1H, s), 11.89 (2H, bs) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	DMSO-d_6 (39.52 ppm)
	Spectral data:	δ 13.3, 15.8, 53.0, 58.6, 110.4, 114.6, 117.1, 136.6, 137.3, 139.2, 149.0, 154.8 ppm
Melting point:		207-210 °C (Decomposition)
Microanalysis:	Found:	C = 50.9%, H = 5.5%, N = 15.0% (August 2006)
	Found:	C = 51.0%, H = 5.2%, N = 15.1% (2003)
	Calculated:	C = 51.2%, H = 5.4%, N = 15.0% (Calculated for $C_{12}H_{15}N_3O_3S$)