Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

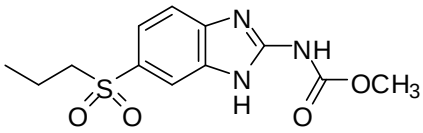
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA P1793: Albendazole sulfone

Report ID: P1793.2020.03

Chemical Formula: $C_{12}H_{15}N_3O_4S$

Molecular Weight: 297.33 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
03-AV-08	76567-28-7	99.6 ± 0.3%

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

IUPAC name: Methyl hydrogen [6-(propylsulfonyl)-1H-benzimidazol-2-yl]carbonimidate.

Expiration of certification: The property values are valid till 20 February 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 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 25 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%. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

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

This report supersedes any issued prior to 13 October 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 qualitative elemental microanalysis.

HPLC:	Instrument:	Waters alliance 2650 or Waters Model 1525 Binary pump, 717 plus autosampler or Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	X-Bridge C-18, 5 μm (4.6 mm x 150 mm)
	Column oven:	70 °C
	Mobile Phase:	A = MilliQ water (0.1 percent formic acid); B = Acetonitrile 0 min 20% B; 0-18 min 20-38% B; 18-19 min 38-90%B; 19-25 min 90%B; 25-28 min 90-25%B; 28-35 min 90%B
	Flow rate:	1.0 mL/min
	Detector:	Waters 2998 PDA or Shimadzu SPD-M20A PDA operating at 280 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, August 2009)
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, July 2014)
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, February 2020)
HPLC:	Instrument:	Waters HPLC 1525 binary pump, W717 autosampler
	Column:	Alltech C-18, 5 µm (4.6 mm × 150 mm)
	Column oven:	Ambient
	Mobile Phase:	Methanol/MilliQ water with 0.1% trifluoroacetic acid (50:50 v/v)
	Flow rate:	1.0 mL/min
	Detector:	Waters 2998 PDA operating at 225 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.8%, s = 0.02% (10 sub samples in duplicate, July 2003)
	Re-analysis:	Mean = 99.5%, s = 0.05% (5 sub samples in duplicate, July 2006)
Karl Fischer analysis:		Moisture content ≤ 0.2% mass fraction (February 2020)
Thermogravimetric analysis:		Volatiles content < 0.1% and non-volatile residue < 0.2% mass fraction (October 2005)
QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Internal standard:	Trioxane (100.0% mass fraction)
	Initial analysis:	Mean (3.75 ppm) = 99.8%, s = 1.01% (5 sub samples, March 2009)

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
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	320 ($M+Na^+$), 298 ($M+H^+$) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Methanol/Chloroform (1:9) Single spot observed, R_f = 0.58
IR:	Instrument:	BioRad FTS 3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3340, 1702, 1653, 1597, 1467, 1429, 1275, 1133, 1109, 1059, 773 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 0.88 (3H), 1.53 (2H, m), 3.19 (2H, m), 3.78 (3H, s), 7.56 – 7.60 (2H, m), 7.89 (1H, s), 12.04 (2H, bs) ppm
^{13}C NMR:	Instrument:	Bruker DMX-300
	Field strength:	75.5MHz
	Solvent:	DMSO-d_6 (39.52 ppm)
	Spectral data:	δ 12.8, 16.8, 53.1, 57.5, 114.4, 121.0, 131.6, 136.2, 141.1, 150.0, 154.6 ppm
Melting point:		> 320 °C
Microanalysis:	Found:	C = 48.5%, H = 5.3%, N = 14.3%, S = 10.8% (August 2006)
	Calculated:	C = 48.5%, H = 5.1%, N = 14.1%, S = 10.8% (Calculated for $C_{12}H_{15}N_3O_4S$)