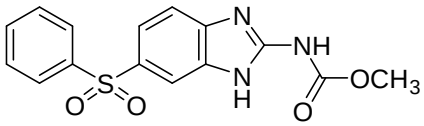
REFERENCE MATERIAL ANALYSIS REPORT

Report ID: P1791.2016.01

Compound Name: **Fenbendazole Sulfone**
Collection Number: P1791
Chemical Formula: $C_{15}H_{13}N_3O_4S$
CAS Number: 54029-20-8
Structure:



Description: Off white powder
Batch Number: 03-AV-06
Molecular Weight: 331.3
Release date: 27th October 2003

Synonyms: [5-(Phenylsulfonyl)-1*H*-benzimidazol-2-yl]carbamic acid methyl ester;
5-(Phenylsulfonyl)-2-benzimidazolecarbamic acid methyl ester;
Methyl-5-(phenylsulfonyl)-2-benzimidazolecarbamate

Purity (mass fraction): 98.6 ± 3.3% (95% coverage interval)

The purity value was obtained from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (QNMR). The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by HPLC with UVdetection, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR. The purity estimate by QNMR was obtained using the three proton singlet at 3.8 ppm, against a certified internal standard of trioxane. Supporting evidence is provided by elemental microanalysis.

QNMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz Solvent: d_6-DMSO
	Internal standard:	Trioxane (100% mass fraction)
	Purity estimate:	Mean (3.8 ppm) = 98.6%, s = 0.4 %, (5 sub samples in duplicate, May 2012)
HPLC:	Column:	Alltech C-18, 5 µm (4.6 mm × 150 mm)
	Column oven:	40 °C
	Mobile Phase:	A: 1.25% TEA in H_2O/MeOH/Acetonitrile (75:15:13) B: Acetonitrile 0-10 min 20% B, 10-11 min 20-80% B, 11-15 min 80% B, 15-16 min 80-20% B, 16-25 min 20% B The aqueous phase (A) was buffered at pH 3.1 using phosphoric acid
	Flow Rate:	1 mL/min
	Detector:	UV at 225 nm (2003 and 2009) and at 294 nm (2012 and 2016)
	Relative peak area of main component :	
	Initial analysis:	Mean = 98.5%, s = 0.03% (7 sub samples in duplicate, July 2003)
	Re-analysis:	Mean = 97.2%, s = 0.07% (5 sub samples in duplicate, August 2009)
	Re-analysis;	Mean = 98.7%, s = 0.01% (5 sub samples in duplicate, April 2012)
	Re-analysis;	Mean = 98.6%, s = 0.02% (5 sub samples in duplicate, February 2016)

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Micromass Quattro Micro
	Operation:	Positive ion mode, direct infusion at 5 μL/min
	Solvent:	MeOH/H_2O with 0.1% CH_3CO_2H
	Ionisation:	ESI spray voltage at 2.8 kV for positive ion mode
	Desolvation temp:	200 °C
	Multiplier:	600 V
	Scan Range:	50-500 m/z
	Peak:	354 ($M\text{-}Na^+$, 7), 332 ($M\text{-}H^+$, 100) m/z
IR:	Instrument:	BioRad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3400, 3356, 3082, 1723, 1643, 1591, 1527, 1460, 1279, 1152, 1088, 764 cm^{-1}
^1H NMR:	Instrument:	Bruker DMX600
	Field strength:	600 MHz Solvent: DMSO-d_6
	Spectral data:	δ 3.77 (3H, s), 7.55-7.65 (5H, m), 7.91 (2H, d), 7.98 (1H, s), 11.87 (2H, bs) ppm
^{13}C NMR:	Instrument:	Bruker DMX300
	Field strength:	75.5 MHz Solvent: DMSO-d_6
	Spectral data:	δ 53.1, 113.9, 120.9, 127.3, 129.9, 133.4, 142.7, 150.2, 154.5 ppm
Melting point:		> 290 °C
Microanalysis:		Found: C = 54.2%; H = 3.8%; N = 12.6% Calc: C = 54.4%; H = 4.0%; N = 12.7% (Calculated for $C_{15}H_{13}N_3O_4S$)

Expiration of certification

The property values are valid till 11th February 2021, i.e. five 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 long-term stability of the compound in solution has not been examined.

This material has demonstrated stability over a minimum period of five years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from annual stability trials.

Homogeneity assessment

The homogeneity of the material was assessed using purity assay by HPLC-UV on seven randomly selected 1-2 mg sub samples of the material. The material was judged to be 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.

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.

Intended Use:

For ***in vitro*** laboratory analysis only.

Caution

Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice

Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 2 March, 2016.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 2nd March 2016.