



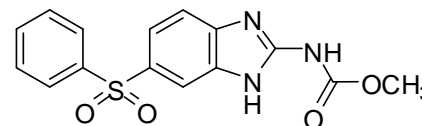
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA P1791: Fenbenazole sulfone

Report ID: P1791.2020.03

Chemical Formula: C<sub>15</sub>H<sub>13</sub>N<sub>3</sub>O<sub>4</sub>S

Molecular Weight: 331.3 g/mol



## Certified value

Batch No.	CAS No.	Purity (mass fraction)
03-AV-06	54029-20-8	98.6 ± 2.3%

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

**IUPAC name:** Methyl [6-(phenylsulfonyl)-1H-benzimidazol-2-yl]carbamate

**Expiration of certification:** The property values are valid till 14 December 2025, 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.

**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 4 °C 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 three 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.

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## Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified purity value was obtained from a combination of traditional analytical techniques and quantitative nuclear magnetic resonance (qNMR). The techniques used in the mass balance approach include HPLC with UV detection, Karl Fischer analysis and <sup>1</sup>H NMR spectroscopy. The purity value is calculated as per Equation 1.

$$\text{Purity} = (100\% - I_{\text{ORG}}) \times (100\% - I_{\text{VOL}} - I_{\text{NVR}}) \quad \text{Equation 1}$$

$I_{\text{ORG}}$  = Organic impurities of related structure,  $I_{\text{VOL}}$  = volatile impurities,  $I_{\text{NVR}}$  = non-volatile residue

The certified purity value by qNMR was obtained using the three-proton singlets at 3.8 ppm measured against a certified internal standard of trioxane.

Supporting evidence is provided by elemental microanalysis.

HPLC:	Instrument:	Waters 2695 Separation module or Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Alltech C-18, 5 µm (4.6 mm × 150 mm)
	Column oven:	40 °C
	Mobile Phase:	A: 1.25% TEA in H <sub>2</sub> O/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, 2016 and 2020)
	Relative mass fraction 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)
	Re-analysis;	Mean = 98.8%, s = 0.03% (5 sub samples in duplicate, December 2020)
	Karl Fischer analysis:	Moisture content 0.20% mass fraction (November 2015 and 2020)
QNMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	DMSO- <i>d</i> <sub>6</sub> (2.50 ppm)
	Internal standard:	Trioxane (100% mass fraction)
	Initial analysis:	Mean (3.8 ppm) = 98.6%, s = 0.4% (5 sub samples, May 2012)

## Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 5 $\mu$ L/min
	Ionisation:	ESI spray voltage at 2.8 kV positive ion
	EM voltage:	600 V
	Cone voltage:	20 V
	Peak:	354 (M+Na <sup>+</sup> ), 332 (M+H <sup>+</sup> ) <i>m/z</i>
IR:	Instrument:	BioRad FTS3000MX FT-IR
	Range:	4000-400 $\text{cm}^{-1}$ , KBr powder
	Peaks:	3400, 3356, 3082, 1723, 1643, 1591, 1527, 1460, 1279, 1152, 1088, 764 $\text{cm}^{-1}$
<sup>1</sup> H NMR:	Instrument:	Bruker DMX600
	Field strength:	600 MHz
	Solvent:	DMSO-d <sub>6</sub> (2.50 ppm)
	Spectral data:	$\delta$ 3.77 (3H, s), 7.55-7.65 (5H, m), 7.91 (2H, d), 7.98 (1H, s), 11.87 (2H, bs) ppm
<sup>13</sup> C NMR:	Instrument:	Bruker DMX300
	Field strength:	75.5 MHz
	Solvent:	DMSO-d <sub>6</sub> (39.5 ppm)
	Spectral data:	$\delta$ 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%
	Calculated:	C = 54.4%; H = 4.0%; N = 12.7% (Calculated for C <sub>15</sub> H <sub>13</sub> N <sub>3</sub> O <sub>4</sub> S)