



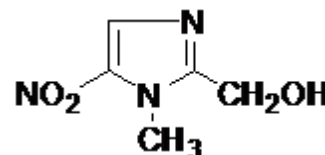
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA P1296: HMMNI

Report ID: P1296.2021.03

Chemical Formula: C<sub>5</sub>H<sub>7</sub>N<sub>3</sub>O<sub>3</sub>

Molecular Weight: 157.1 g/mol



### Certified value

Batch No.	CAS No.	Purity (mass fraction)
96-014679	936-05-0	99.5 ± 0.3%

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

**IUPAC name:** (1-Methyl-5-nitro-1H-imidazol-2-yl)methanol.

**Expiration of certification:** The property values are valid till 20 August 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:** Pale yellow crystals 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 °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%.

**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 GC-FID on five 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.  
16 August 2022

This report supersedes any issued prior to 16 August 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 by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, 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.

Supporting evidence is provided by elemental microanalysis.

GC-FID:	Instrument:	HP5890
	Column:	ZB-1 capillary, 30 m x 0.32 mm I.D. x 0.25 µm
	Program:	100 °C (1 min), 10 °C/min to 250 °C, 40 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.1% (7 sub samples, March 1996)
	Re-analysis:	Mean = 99.9%, s = 0.005% (5 sub samples in duplicate, September 2006)
GC-FID:	Instrument:	Varian 3800 or Agilent 8890
	Column:	HP-1MS capillary, 30 m x 0.32 mm I.D. x 0.25 µm
	Program:	100 °C (1 min), 10 °C/min to 250 °C (1 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, September 2011)
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, August 2021)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (August 2006, September 2011 and June 2021)
Thermogravimetric analysis:		Volatiles content < 0.1% and non-volatile residue < 0.2% mass fraction. (August 2006)

**Spectroscopic and other characterisation data**

GC-MS: Instrument: HP5890/5970B  
Column: HP Ultra-2, 12 m x 0.22 I.D x 0.11  $\mu$ m  
Program: 70 °C (1 min), 20 °C/min to 300 °C (3 min)  
Injector: 230 °C  
Split ratio: 20/1  
Transfer line temp: 280 °C  
Carrier: Helium  
Scan range: 50-550 *m/z*

The retention times of the parent compound and *bis*-TMS derivative are reported with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.

Parent (8.7 min): 157 ( $M^+$ ), 140, 128, 127, 111, 94, 81, 70, 54, 42 *m/z*  
*Bis*-TMS (13.9 min): 229 ( $M^+$ ), 214, 184, 197, 184, 168, 167, 153, 140, 129, 99, 84, 73 *m/z*

TLC: Conditions: Kieselgel 60F<sub>254</sub>. Diisopropylether/diethylether/diethylamine (45/45/10)  
Single spot observed,  $R_f = 0.10$

IR: Instrument: FT-IR, Biorad WIN FTS40  
Range: 4000-400  $\text{cm}^{-1}$  (KBr pellet)  
Peaks: 1531, 1474, 1385, 1364, 1269, 1238, 1188, 1041, 831  $\text{cm}^{-1}$

<sup>1</sup>H NMR: Instrument: Bruker DMX 600  
Field strength: 600 MHz  
Solvent: D<sub>2</sub>O (4.79 ppm)  
Spectral data:  $\delta$  3.94 (3H, s), 4.73 (2H, s), 8.00 (1H, s) ppm  
Acetone estimated at 0.3% mass fraction was observed in the <sup>1</sup>H NMR

<sup>13</sup>C NMR: Instrument: Bruker Avance III-500  
Field strength: 126 MHz  
Solvent: D<sub>2</sub>O  
Spectral data:  $\delta$  33.2, 55.6, 131.5, 139.4, 151.2 ppm

Melting point: 115-117°C

Microanalysis: Found: C = 38.5%; H = 4.6%; N = 26.9% (August, 2006)  
Calculated: C = 38.2%; H = 4.5%; N = 26.7% (Calculated for C<sub>5</sub>H<sub>7</sub>N<sub>3</sub>O<sub>3</sub>)