



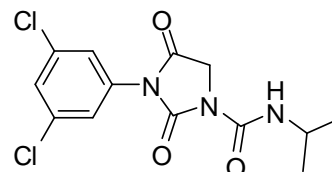
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA P1805: Iprodione

Report ID: P1805.2017.04

Chemical Formula: C<sub>13</sub>H<sub>13</sub>Cl<sub>2</sub>N<sub>3</sub>O<sub>3</sub>

Molecular Weight: 330.2 g/mol



## Certified value

Batch No.	CAS No.	Purity (mass fraction)
10-AV-02	36734-19-7	99.7 ± 0.6%

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

**IUPAC name:** 3-(3,5-Dichlorophenyl)-N-isopropyl-2,4-dioxo-1-imidazolidinecarboxamide.

**Expiration of certification:** The property values are valid till 7 April 2020, i.e. three 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 material will be re-tested on an annual basis to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

**Description:** 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 3 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 ten 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.  
18 October 2022

This report supersedes any issued prior to 18 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 GC-FID/HPLC with UV/ELS detection 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

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The purity value by qNMR was obtained using a combination of the six proton doublets at 1.2 ppm, the one-proton multiplet at 4.0 ppm and the two-proton singlet at 4.4 ppm measured against a certified internal standard of dimethyl sulfone.

Supporting evidence is provided by qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A autosampler	
	Column:	Grace Alltima C-18, 5 µm (4.6 mm x 150 mm)	
	Column oven:	40 °C	
	Mobile Phase:	Acetonitrile/MilliQ water (50:50 v/v)	
	Flow rate:	1.0 mL/min	
	Detector:	Shimadzu SPD-M20A PDA operating at 220 nm	
	Relative mass fraction of the main component:		
	Initial analysis:	Mean = 99.9%, s = 0.01% (10 sub samples in duplicate, March 2011)	
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, March 2012)	
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, February 2013)	
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, February 2014)	
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, April 2017)	
Karl Fischer analysis:		Moisture content < 0.2% mass fraction (January 2011)	
		Moisture content <0.1% mass fraction (March 2012, March 2013, February 2014 and April 2017)	
Thermogravimetric analysis:		Non volatile residue < 0.2% mass fraction (January 2011). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material.	
QNMR:	Instrument:	Bruker Avance- 500	
	Field strength:	500 MHz	Solvent: CDCl <sub>3</sub> (7.26 ppm)
	Internal standard:	Dimethyl sulfone (100.0% mass fraction)	
	Initial analysis:	Mean = 99.6%, s = 0.3% (5 sub samples, January 2011)	

**Spectroscopic and other characterisation data**

GC-MS: Instrument: Agilent 6890/5973  
Column: TG-1MS, 30 m x 0.25 mm I.D. x 0.25  $\mu$ m  
Program: 180 °C (1 min), 10 °C/min to 300 °C (3 min)  
Injector: 250 °C  
Split ratio: 20/1  
Transfer line temp: 280 °C  
Carrier: Helium  
Scan range: 50-550  $m/z$

The retention time of the parent compound is 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 (9.4 min): 331 ( $M^+$ , 2), 329 ( $M^+$ , 3), 316 (66), 314 (100), 245 (32), 243 (47), 189 (54), 186 (82), 124 (22), 70 (53), 56 (33)  $m/z$

HS-GC-MS: Instrument: Agilent 6890/5973/G1888  
Column: DB-624, 30 m x 0.25 mm I.D. x 1.4  $\mu$ m  
Program: 50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min)  
Injector: 150 °C  
Transfer line temp: 280 °C  
Carrier: Helium, 1.2 mL/min  
Split ratio: 50/1  
Solvents detected: Diisopropyl ether

TLC: Conditions: Kieselgel 60F<sub>254</sub>. Chloroform/Ethyl acetate (9:1)  
Single spot observed,  $R_f = 0.70$

IR: Instrument: Biorad FTS3000MX FT-IR  
Range: 4000-400  $\text{cm}^{-1}$ , KBr powder  
Peaks: 3353, 3087, 2975, 2936, 2877, 1779, 1727, 1574, 1542, 1450, 1396, 1278, 1240, 1202, 1143, 857, 804, 742  $\text{cm}^{-1}$

<sup>1</sup>H NMR: Instrument: Bruker Avance III-400  
Field strength: 400 MHz  
Solvent: CDCl<sub>3</sub> (7.26 ppm)  
Spectral data:  $\delta$  1.23 (6H, d,  $J = 6.6$  Hz), 4.06 (1H, octet,  $J = 6.4$  Hz), 4.44 (2H, s), 7.27 (2H, d,  $J = 1.8$  Hz), 7.42 (1H, t,  $J = 1.8$  Hz) 7.58 (1H, d,  $J = 7.5$  Hz) ppm  
Diisopropyl ether estimated at 0.06% mass fraction was observed in the <sup>1</sup>H NMR

<sup>13</sup>C NMR: Instrument: Bruker Avance III-400  
Field strength: 100 MHz  
Solvent: CDCl<sub>3</sub> (77.0 ppm)  
Spectral data:  $\delta$  22.7, 42.8, 47.8, 124.5, 129.0, 132.2, 135.5, 149.4, 153.7, 166.2 ppm

Melting point: 133-134 °C

Microanalysis: Found: C = 47.6%; H = 3.8%; N = 12.9%; Cl = 21.4% (February, 2011)  
Calculated: C = 47.3%; H = 4.0%; N = 12.7%; Cl = 21.5%  
(Calculated for C<sub>13</sub>H<sub>13</sub>Cl<sub>2</sub>N<sub>3</sub>O<sub>3</sub>)