Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

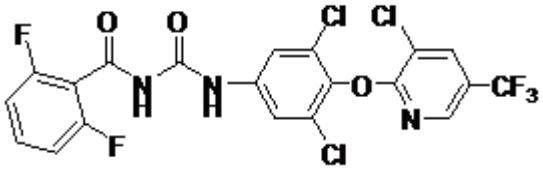
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA P1489: Chlorfluazuron

Report ID: P1489.2020.03

Chemical Formula: $C_{20}H_9Cl_3F_5N_3O_3$

Molecular Weight: 540.7 g/mol



Property value

Batch No.	CAS No.	Purity estimate
97-001732	71422-67-8	98.8%

IUPAC name: N-[(3,5-Dichloro-4-{[3-chloro-5-(trifluoromethyl)-2-pyridinyl]oxy}phenyl)carbamoyl]-2,6-difluorobenzamide.

Expiration of certification: The property values are valid till 17 August 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: White crystals 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 reference material should be used for qualitative analysis only.

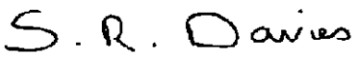
Instructions for use: Equilibrate the bottled material to room temperature before opening.

Recommended storage: When not in use this material should be stored at or below 4 ºC in a closed container in a dry, dark area.

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 HPLC with UV detection 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.



Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
22 August 2022

This report supersedes any issued prior to 22 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.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The 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.

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

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

Supporting evidence is provided by elemental microanalysis.

HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler or Water alliance 2695
	Column:	Alltima C-18, 5 μm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	A = MilliQ water; D = Acetonitrile 0-10 min 70% D; 10-11 min 70-90% D; 11-15 min 90% D, 15-16 min 90-70% D; 16-22 min 70% D
	Flow rate:	1.0 mL/min
	Detector:	Waters 2998 PDA operating at 260 nm
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.6%, s = 0.4% (7 sub samples in duplicate, September 1997)
	Re-analysis:	Mean = 99.1%, s = 0.02% (5 sub samples in duplicate, June 2008)
	Re-analysis:	Mean = 99.0%, s = 0.01% (5 sub samples in duplicate, May 2013)
	Re-analysis:	Mean = 99.1%, s = 0.01% (5 sub samples in duplicate, August 2020)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (June 2008, April 2013 and July 2020)

Spectroscopic and other characterisation data

^{1}H NMR:	Instrument:	Bruker ACF 300 (300 MHz)
	Field strength:	300 MHz
	Solvent:	$CDCl_3$ + TFA
	Spectral data:	δ 7.10 (2H, m), 7.53-7.65 (1H, m), 7.70 (2H, s), 8.10 (1H, d), 8.30 (1H, m) ppm Acetone observed (2.3 ppm) at about 0.2% mass fraction
^{13}C NMR:	Instrument:	Bruker ARX-500
	Field strength:	125 MHz
	Solvent:	$CDCl_3$ + TFA
	Spectral data:	δ 110.8, 112.7, 113.0, 115.3, 117.6, 119.8, 121.4, 121.5, 129.6, 134.9, 137.8, 141.8, 142.6, 152.5, 159.0, 159.4, 160.3 ppm
TLC:	Conditions:	Kieselgel 60F_{254}. toluene/acetone/ethanol/ammonia (45/45/7/3) Single spot observed, R_f = 0.73
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400 cm^{-1}, KBr plate
	Peaks:	1719, 1595, 1553, 1471, 1450, 1400, 1323, 1293, 1271, 1235, 1210, 1163, 1138 cm^{-1}
Microanalysis:	Found:	C = 44.6%, H = 1.7% (June 2008)
	Calculated:	C = 44.4%, H = 1.7% (Calculated for $C_{20}H_9Cl_3F_5N_3O_3$)