Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

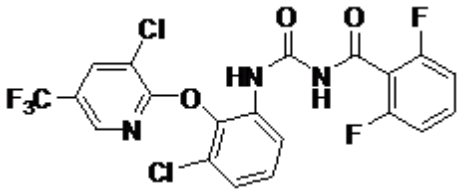
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA P1490: Fluazuron

Report ID: P1490.2020.03

Chemical Formula: $C_{20}H_{10}Cl_2F_5N_3O_3$

Molecular Weight: 506.2 g/mol



## Certified value

| Batch No. | CAS No. | Purity (mass fraction) |
|---|---|---|
| 97-000666 | 86811-58-7 | 95.6 ± 2.2% |

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

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

**Expiration of certification:** The property values are valid till 21 September 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:** White solid 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 25 ºC 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 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.
6 September 2022

This report supersedes any issued prior to 6 September 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 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 thermogravimetric analysis, Karl Fischer analysis and $^{1}$H NMR spectroscopy. The purity value is calculated as per Equation 1.

Purity = (100 % - $I_{ORG}$) x (100 % - $I_{VOL}$ – $I_{NVR}$) Equation 1

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

The certified purity value by qNMR was obtained using a combination of the one proton triplet at 7.0 ppm, the one proton doublet at 8.0 ppm and the one proton doublet at 8.3 ppm 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: | Waters Model 1525 Binary pump, 717 plus autosampler |
| | Column: | Alltech Alltima C18, 5 µm (4.6 mm. x 150 mm). |
| | Column oven: | Ambient |
| | Mobile Phase: | Acetonitrile/MilliQ water (3:1 v/v) |
| | Flow rate: | 1.0 mL/min |
| | Detector: | Waters 2998 PDA operating at 260 nm |
| | Relative mass fraction of the main component: | |
| | Initial analysis: | Mean = 99.9%, s = 0.1% (7 sub samples in duplicate, April 1999) |
| | Re-analysis: | Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, August 2005) |
| | Re-analysis: | Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, September 2020) |
| HPLC: | Instrument: | Waters Model 1525 Binary pump, 717 plus autosampler |
| | Column: | Ascentis C-18, 2.7 µm (4.6 mm x 150 mm) |
| | Column oven: | Ambient |
| | Mobile Phase: | Acetonitrile/MilliQ water (65:35 v/v) |
| | Flow rate: | 1.0 mL/min |
| | Detector: | Waters 2998 PDA operating at 260 nm |
| | Relative mass fraction of the main component: | |
| | Initial analysis: | Mean = 99.9%, s = 0.002% (5 sub samples in duplicate, November 2010) |
| Thermogravimetric analysis: | | Volatile content 4.0% and non volatile content < 0.2% mass fraction (October 2005 and July 2006)<br>Volatile content 4.4% and non volatile residue < 0.1% mass fraction (November 2010)<br>Volatile content 3.4% and non volatile residue < 0.1% mass fraction (October 2020) |
| QNMR: | Instrument: | Bruker DMX-600 |
| | Field strength: | 600 MHz |
| | Solvent: | $CDCl_3$ (7.26 ppm) |
| | Internal standard: | Dimethyl sulfone (100% mass fraction) |
| | Initial analysis: | Mean (7.0 ppm) = 95.6%, s = 0.4% (5 sub samples, December 2011)<br>Mean (8.0 ppm) = 95.1%, s = 0.6% (5 sub samples, December 2011)<br>Mean (8.3 ppm) = 95.2%, s = 0.5% (5 sub samples, December 2011) |

## Spectroscopic and other characterisation data

| | | |
|---|---|---|
| TLC: | Conditions: | Kieselgel 60$F_{254}$. toluene/acetone/diethylamine (85:10:5)<br>Single spot observed, $R_f$ = 0.5 |
| IR: | Instrument:<br>Range:<br>Peaks: | Bruker Alpha Platinum ATR<br>4000-400 $cm^{-1}$,neat<br>1723, 1699, 1603, 1558, 1466, 1329, 1236, 1071, 1018, 794 $cm^{-1}$ |
| $^1$H NMR: | Instrument:<br>Field strength:<br>Solvent:<br>Spectral data: | Bruker Avance III-500<br>500 MHz<br>$CDCl_3$ + TFA (7.26 ppm)<br>$\delta$ 7.10 (2H, m), 7.40 (1H, dd), 7.50-7.65 (2H, m), 8.15 (1H, d), 8.35 (1H, m) ppm |
| Melting point: | | 217-219 °C |
| Microanalysis: | Found:<br>Found:<br>Calculated: | C = 46.5%; H = 2.0%; N = 8.1%, F = 18.4%, Cl = 16.8% (December 2011)<br>C = 46.3%; H = 2.0%; N = 8.1% (August 2005)<br>C = 47.5%; H = 2.0%; N = 8.3%, F = 18.8%, Cl = 14.0% (Calc for $C_{20}H_{10}Cl_2F_5N_3O_3$) |