



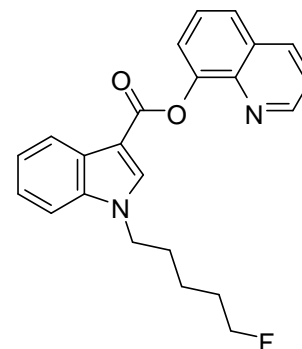
# REFERENCE MATERIAL PRODUCT INFORMATION SHEET

## NMIA D1023: Quinolin-8-yl 1-(5-fluoropentyl)-1H-indole-3-carboxylate

Report ID: D1023.2020.03

Chemical Formula: C<sub>23</sub>H<sub>21</sub>FN<sub>2</sub>O<sub>2</sub>

Molecular Weight: 376.4 g/mol



### Property value

Batch No.	CAS No.	Purity estimate
14-D-22	1400742-41-7	99.3 ± 1.9%

**IUPAC name:** 8-Quinoliny 1-(5-fluoropentyl)-1H-indole-3-carboxylate.

**Expiration of certification:** The property values are valid till 29 July 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:** 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 reference material is recommended 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 25 °C in a closed container in a dry, dark area.

**Stability:** In the absence of long term stability data the stability of this material has been judged from stability trials conducted on similar materials by NMI Australia over the last ten years. This material has demonstrated stability over a minimum period of three years. 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.  
21 September 2022

This report supersedes any issued prior to 21 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.

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**Characterisation Report:**

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. Impurities of related structure were assessed by HPLC with UV detection. The purity value was obtained by mass balance from a combination of traditional analytical techniques, including HPLC with UV detection. 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 Karl Fischer analysis,  $^1\text{H}$  NMR spectroscopy and elemental microanalysis.

HPLC: Instrument: Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler or Water alliance 2695  
Column: Grace Alltima C-18, 5  $\mu\text{m}$  (4.6 mm x 150 mm)  
Column oven: 35°C or 40°C  
Mobile Phase: Acetonitrile/MilliQ water (65:35 v/v)  
Flow rate: 0.8 mL/min or 1 mL/min  
Detector: Shimadzu SPD-M20A PDA or Waters 2998 PDA operating at 300 nm  
Relative peak area of the main component:  
Initial analysis: Mean = 99.8%, s = 0.01% (10 sub samples in duplicate, November 2014)  
Re-analysis: Mean = 99.8%, s = 0.002% (5 sub samples in duplicate, October 2015)  
Re-analysis: Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, October 2016)  
Re-analysis: Mean = 99.8%, s = 0.004% (5 sub samples in duplicate, September 2017)  
Re-analysis: Mean = 99.8%, s = 0.004% (5 sub samples in duplicate, July 2020)

Karl Fischer analysis: Moisture content < 0.2% mass fraction (November 2014, October 2015, September 2016, August 2017 and July 2020)

Thermogravimetric analysis: Non-volatile residue < 0.2% mass fraction (November 2014)

## Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890
	Column:	TG-5MS, 30 m x 0.25 mm I.D. x 0.25 $\mu$ m
	Program:	180 °C (1 min), 30 °C/min to 300 °C (20 min)
	Injector:	250 °C,
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	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 (11.7 min):	376 ( $M^+$ , 3), 232 (100), 144 (17), 116 (8), 89 (5), 63 (1), 41 (2) $m/z$
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 $\mu$ L/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	10 V
	Peak:	377.3 ( $M+H^+$ ) $m/z$
TLC:	Conditions:	Kieselgel 60F <sub>254</sub> . Hexane/ethyl acetate (16:9) Single spot observed, $R_f = 0.29$ . Visualisation with UV at 254 nm
IR:	Instrument:	Bruker Alpha FT-IR
	Range:	4000-400 $cm^{-1}$ , neat
	Peaks:	1713, 1527, 1463, 1380, 1192, 1166, 1083, 964, 875, 811, 779, 738 $cm^{-1}$
<sup>1</sup> H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	<i>d</i> <sub>6</sub> -DMSO (2.50 ppm)
	Spectral data:	$\delta$ 1.39 (2H, quintet, $J = 7.5$ Hz), 1.67-1.75 (2H, m), 1.89 (2H, quintet, $J = 7.5$ Hz), 4.37 (2H, t, $J = 7.5$ Hz), 4.43 (2H, dt, $J_{H-F} = 48$ Hz, $J_{H-H} = 6.0$ Hz), 7.26 (1H, t, $J = 7.9$ Hz), 7.32 (1H, t, $J = 7.2$ Hz), 7.59 (1H, dd, $J = 4.2, 8.4$ Hz), 7.66-7.68 (3H, m), 7.70 (1H, bd, $J = 8.2$ Hz), 7.94 (1H, m), 8.06 (1H, d, $J = 7.7$ Hz), 8.46 (1H, dd, $J = 1.7, 8.4$ Hz), 8.50 (1H, s), 8.85 (1H, dd, $J = 1.6, 4.0$ Hz) ppm Ethyl acetate and hexane were observed in the <sup>1</sup> H NMR at an estimated 0.3 and 0.2% mass fraction respectively
<sup>13</sup> C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	<i>d</i> <sub>6</sub> -DMSO (39.5 ppm)
	Spectral data:	$\delta$ 22.1 ( $J_{CF} = 5.3$ Hz), 29.3, 29.4 ( $J_{CF} = 19.3$ Hz), 46.1, 83.7 ( $J_{CF} = 161.7$ Hz), 104.6, 111.2, 120.8, 121.9, 122.05, 122.13, 122.7, 125.9, 126.50, 126.51, 129.2, 136.2, 136.5, 136.6, 141.2, 147.3, 150.5, 162.5 ppm
<sup>19</sup> F NMR:	Instrument:	Bruker Avance III-500
	Field strength:	376 MHz
	Solvent:	<i>d</i> <sub>6</sub> -DMSO
	Spectral data:	$\delta$ -216.9 ppm
Melting point:		117-119 °C
Microanalysis:	Found:	C = 73.5%; H = 5.6%; N = 7.5%; F = 5.2% (November, 2014)
	Calculated:	C = 73.4%; H = 5.6%; N = 7.4%; F = 5.1% (Calculated for C <sub>23</sub> H <sub>21</sub> FN <sub>2</sub> O <sub>2</sub> )