



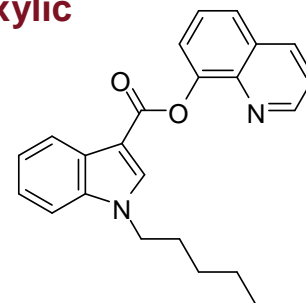
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA D1022: 1-Pentyl-8-quinolinyl ester-1H-indole-3-carboxylic acid

Report ID: D1022.2020.03

Chemical Formula: C<sub>23</sub>H<sub>22</sub>N<sub>2</sub>O<sub>2</sub>

Molecular Weight: 358.4 g/mol



### Certified value

Batch No.	CAS No.	Purity (mass fraction)
14-D-21	1400742-17-7	99.7 ± 0.4%

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

**IUPAC name:** 8-Quinolinyl-1-pentyl-1H-indole-3-carboxylate.

**Expiration of certification:** The property values are valid till 20 August 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:** 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 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 °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 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 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. The certified 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 <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 qualitative elemental microanalysis.

HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler or Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	Alltima C-18, 5 $\mu\text{m}$ (4.6 mm x 150 mm)
	Column oven:	40 $^{\circ}\text{C}$
	Mobile Phase:	Acetonitrile/MilliQ water (75:25)
	Flow rate:	0.8 mL/min
	Detector:	Waters 2998 PDA operating at 300 nm or Shimadzu SPD-M20A PDA operating at 300 nm

Relative mass fraction of the main component:

Initial analysis:	Mean = 99.8%, s = 0.01% (10 sub samples in duplicate, July 2014)
Re-analysis:	Mean = 99.8%, s = 0.02% (5 sub samples in duplicate, August 2015)
Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, August 2016)
Re-analysis:	Mean = 99.7%, s = 0.01% (5 sub samples in duplicate, July 2017)
Re-analysis:	Mean = 99.7%, s = 0.02% (5 sub samples in duplicate, August 2020)

Karl Fischer analysis: Moisture content  $\leq$  0.1% mass fraction (July 2014, 2015, 2016, 2017 and 2020)

Thermogravimetric analysis: Volatiles content < 0.1% and non-volatile residue < 0.2% mass fraction (July 2014)

## Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	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), 30 °C/min to 300 °C (20 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium
	Scan range:	50-550 <i>m/z</i>
	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 (10.6 min):	358 (M+, 3), 214 (100), 191 (< 4), 144 (16) 116 (8), 89 (5) <i>m/z</i>
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:	8 V
	Peak:	359.2 (M+H <sup>+</sup> ) <i>m/z</i>
TLC:	Conditions:	Kieselgel 60F <sub>254</sub> . Hexane/ethyl acetate (3:2) Single spot observed, R <sub>f</sub> = 0.6
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm <sup>-1</sup> , KBr powder
	Peaks:	1707, 1528, 1467, 1379, 1184, 1105, 1083, 965, 848, 812, 752 cm <sup>-1</sup>
<sup>1</sup> H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	DMSO- <i>d</i> <sub>6</sub> (2.50 ppm)
	Spectral data:	$\delta$ 0.86 (3H, t, <i>J</i> = 7.2 Hz), 1.25-1.35 (4H, m), 1.84 (2H, quintet, <i>J</i> = 7.1 Hz), 4.34 (2H, t, <i>J</i> = 7.1 Hz), 7.26 (1H, dt, <i>J</i> = 0.8, 7.9 Hz), 7.32 (1H, dt, <i>J</i> = 1.2, 7.2 Hz), 7.58 (1H, dd, <i>J</i> = 4.2, 8.4 Hz), 7.66-7.67 (3H, m), 7.94 (1H, m), 8.06 (1H, d, <i>J</i> = 7.7 Hz), 8.46 (1H, dd, <i>J</i> = 1.6, 8.4 Hz), 8.49 (1H, s), 8.85 (1H, dd, <i>J</i> = 1.6, 4.0 Hz) ppm Dichloromethane estimated at 0.05% 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:	DMSO- <i>d</i> <sub>6</sub> (39.52 ppm)
	Spectral data:	$\delta$ 13.9, 21.8, 28.4, 29.3, 46.3, 104.6, 111.2, 120.8, 121.9, 122.0, 122.1, 122.7, 125.8, 126.5, 129.2, 136.2, 136.5, 136.6, 141.2, 147.3, 150.5, 162.5 ppm
Melting point:	112-114 °C followed by 122-123 °C	
Microanalysis:	Found:	C = 76.9%; H = 6.2%; N = 7.8% (July, 2014)
	Calculated:	C = 77.2%; H = 6.2%; N = 7.8%; (Calculated for C <sub>23</sub> H <sub>22</sub> N <sub>2</sub> O <sub>2</sub> )