



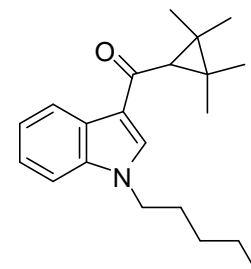
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D1014: (1-Pentyl-1H-indol-3-yl)-(2,2,3,3-tetramethylcyclopropyl)-methanone

Report ID: D1014.2020.03

Chemical Formula: C₂₁H₂₉NO

Molecular Weight: 311.5 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
14-D-09	1199943-44-6	99.6 ± 0.3%

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

IUPAC name: (1-Pentyl-1H-indol-3-yl)(2,2,3,3-tetramethylcyclopropyl)methanone.

Expiration of certification: The property values are valid till 8 May 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 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.

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 ¹H 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_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

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

GC-FID: Instrument: Varian CP-3800
Column: VF-1ms, 30 m × 0.32 mm I.D. × 0.25 μm
Program: 120 °C (1 min), 12 °C/min to 300 °C (3 min)
Injector: 250 °C
Detector Temp: 320 °C
Carrier: Helium
Split ratio: 20/1

Relative peak area of the main component:

Initial analysis: Mean = 99.9%, s = 0.01% (10 sub samples in duplicate, May 2014)

HPLC: Instrument: Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
Column: Ascentis C-18, 5 μm (4.6 mm × 150 mm)
Column oven: 40 °C
Mobile Phase: Acetonitrile/MilliQ water (75:25 v/v)
Flow rate: 1.0 mL/min
Detector: Shimadzu SPD-M20A PDA operating at Max plot

Relative mass fraction of the main component:

Re-analysis: Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, May 2020)

Karl Fischer analysis: Moisture content ≤ 0.2% mass fraction (May 2014)
Moisture content ≤ 0.1% mass fraction (May 2015, 2016, April 2017 and May 2020)

Thermogravimetric analysis: Non-volatile residue 0.2% mass fraction (May 2014). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material and/or degradation at elevated temperatures.

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 μ m
	Program:	60 °C (1 min), 10 °C/min to 100 °C, 15 °C/min to 250 °C (5 min), 30 °C/min to 300 °C (2 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	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 (18.4 min):	311 (M^+ , 31), 296 (36), 252 (17), 229 (16), 214 (100), 144 (33) <i>m/z</i>
ESI-MS:	Instrument:	Micromass Quatro LC Micro
	Operation:	Positive ion mode, direct infusion at 10 μ L/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	20 V
	Peak:	312.3 ($M+H^+$) <i>m/z</i>
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 μ 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:	Hexane, petroleum ether
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Hexane/dichloromethane (1:1) Single spot observed, $R_f = 0.3$
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1} , KBr powder
	Peaks:	3105, 2953, 2930, 1626, 1522, 1466, 1394, 1220, 1201, 1137, 1105, 961, 817 cm^{-1}
¹ H NMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	CDCl ₃ (7.26 ppm)
	Spectral data:	δ 0.91 (3H, t, $J = 6.9$ Hz), 1.26-1.45 (4H, m), 1.31 (6H, s), 1.35 (6H, s), 1.89 (2H, quintet, $J = 7.4$ Hz), 1.95 (1H, s), 4.15 (2H, t, $J = 7.2$ Hz), 7.23-7.30 (2H, m), 7.35 (1H, m), 7.66 (1H, s), 8.41 (1H, m) ppm
¹³ C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	101 MHz
	Solvent:	CDCl ₃ (77.2 ppm)
	Spectral data:	δ 14.1, 17.2, 22.4, 24.2, 29.2, 29.8, 31.7, 41.8, 47.1, 109.8, 119.8, 122.2, 122.8, 123.0, 126.5, 133.6, 136.8, 194.8 ppm
Melting point:		69-70 °C
Microanalysis:	Found:	C = 81.0%; H = 9.5%; N = 4.5% (November, 2015)
	Calculated:	C = 81.0%; H = 9.4%; N = 4.5% (Calculated for C ₂₁ H ₂₉ NO)