Australian Government
Department of Industry, Science and Resources

National Measurement Institute

CIPM MRA

NATA
WORLD RECOGNISED ACCREDITATION

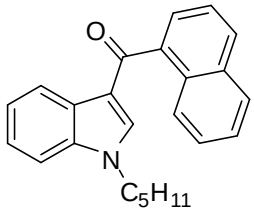
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D955: N-Pentyl-3-(1-naphthoyl)indole

Report ID: D955.2023.01

Chemical Formula: $C_{24}H_{23}NO$

Molecular Weight: 341.5 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
10-D-07	**209414-07-3**	**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-Naphthyl(1-pentyl)-1H-indol-3-yl)-methanone
Synonym: JWH-018

Expiration of certification: The property values are valid till 25 July 2028, 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 powder prepared by synthesis 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 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 GC-FID 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.
9 August 2023

This report supersedes any issued prior to 9 August 2023.

NATA Accreditation No. 198 / Corporate Site No. 14214.

CIPM MRA notice: This certificate is consistent with the capabilities that are included in Appendix C of the CIPM MRA drawn up by the CIPM. Under the CIPM MRA, all participating institutes recognize the validity of each other's calibration and measurement certificates for the quantities, ranges and measurement uncertainties specified in Appendix C. The "CIPM MRA Logo" and this statement attest only to the measurement(s) applied for determining the certified values on the certificate (for details see http://www.bipm.org).

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 GC-FID, 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.

Supporting evidence is provided by qualitative headspace GC-MS analysis of occluded solvents, qNMR, HPLC with UV detection and elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890 or 7890
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	270 °C (12 min), 20 °C/min to 300°C (5 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.01% (10 sub samples in duplicate, July 2010)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, September 2013)
	Re-analysis:	Mean = 99.9%, s = 0.005% (5 sub samples in duplicate, July 2016)
	Re-analysis:	Mean = 99.9%, s = 0.003% (5 sub samples in duplicate, June 2019)
	Re-analysis:	Mean = 99.9%, s = 0.02% (5 sub samples in duplicate, July 2023)
GC-FID:	Instrument:	Varian 3800
	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	270 °C (15 min), 20 °C/min to 300 °C (5 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.9%, s = 0.02% (10 sub samples in duplicate, July 2010)
HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Alltima C-18, 5 μm (4.6 mm x 150 mm)
	Mobile Phase:	Methanol/MilliQ Water (82:18) The aqueous phase was buffered at pH 4.2 using 20 mM NH_4OAc and AcOH
	Flow rate:	1.0 mL/min
	Detector:	Waters PDA 996 operating at Max plot
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.8%, s = 0.1% (10 sub samples in duplicate, September 2010)
Karl Fischer analysis:		Moisture content 0.1% mass fraction (July 2010) Moisture content 0.2% mass fraction (September 2013) Moisture content <0.1% mass fraction (July 2016, May 2019) Moisture content 0.1% mass fraction (August 2023)
Thermogravimetric analysis:		Volatile content 0.30% and non volatile residue < 0.1 % mass fraction (July 2010)
QNMR:	Instrument:	Bruker Avance-600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Internal standard:	Dimethylsulfone (100% m/m)
	Initial analysis:	Mean = 100.2%, s = 0.5% (4 sub samples, September 2010)

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 μm
	Program:	250 °C (15 min), 10 °C/min to 300 °C (5 min)
	Injector:	250 °C
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	30/1
	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 (18.9 min):	341 (M^+, 100), 324 (46), 284 (56), 270 (23), 254 (12), 241 (11), 214 (52), 167 (11), 155 (19), 144 (24), 127 (36) *m/z*
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 μm
	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:	Dichloromethane, ethyl acetate, hexane, cyclohexane, benzene, 3-methyl pentane, methyl cyclopentane
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/methanol (9/1) Single spot observed, R_f = 0.8. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-500 cm^{-1}, KBr powder
	Peaks:	3098, 3052, 2954, 2936, 2866, 1616, 1522, 1465, 1393, 1375, 1231, 1194, 1133, 889, 792, 753 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance 400
	Field strength:	400 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 0.86 (3H, t, *J* = 7.0 Hz), 1.21-1.36 (4H, m), 1.81 (2H, m), 4.06 (2H, t, *J* = 7.2 Hz), 7.34-7.42 (4H, m), 7.45-7.55 (3H, m), 7.67 (1H, dd, *J* = 1.2, 7.0 Hz), 7.92 (1H, m), 7.97 (1H, m), 8.21 (1H, m), 8.49-8.53 (1H, m) ppm Dichloromethane and ethyl acetate estimated at 0.02% and 0.06% mass fraction respectively were observed in the ^{1}H NMR.
^{13}C NMR:	Instrument:	Bruker Avance 400
	Field strength:	100 MHz
	Solvent:	$CDCl_3$ (77.16 ppm)
	Spectral data:	δ 14.0, 22.3, 29.0, 29.6, 47.3, 110.1, 117.7, 122.97, 123.05, 123.7, 124.7, 125.9, 126.1, 126.4, 126.9, 127.1, 128.3, 130.1, 130.9, 133.9, 137.2, 138.1, 139.2, 192.1 ppm
Melting point:		58-63 °C
Microanalysis:	Found:	C = 84.6%; H = 7.0%; N = 4.1% (July 2010)
	Calculated	C = 84.4%; H = 6.8%; N = 4.1% (Calculated for $C_{24}H_{23}NO$)