



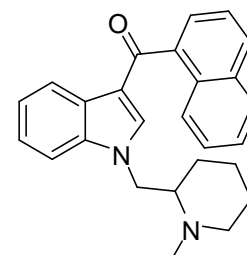
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA D982: 1-[(1-Methylpiperidin-2-yl) methyl]-3-(1-naphthoyl)-indole (AM1220)

Report ID: D982.2021.02

Chemical Formula: C<sub>26</sub>H<sub>26</sub>N<sub>2</sub>O

Molecular Weight: 382.5 g/mol



### Certified value

Batch No.	CAS No.	Purity (mass fraction)
12-D-10	137642-54-7	99.0 ± 2.0%

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

**IUPAC name:** [1-[(1-Methyl-2-piperindinyl)methyl]-1H-indol-3-yl]-1-naphthalenyl-methanone

**Expiration of certification:** The property values are valid till 16 November 2026, 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. This material has been given a shelf life of three years from the date of re-certification.

**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%. 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 five 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.  
20 September 2022

This report supersedes any issued prior to 20 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 by qNMR was obtained using the one-proton doublet of doublets at 4.5 ppm measured against a certified internal standard of 1,4-bis(trimethylsilyl)benzene.

$$\text{Purity} = (100\% - I_{\text{ORG}}) \times (100\% - I_{\text{VOL}} - I_{\text{NVR}}) \quad \text{Equation 1}$$

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

Supporting evidence is provided by a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis,  $^1\text{H}$  NMR spectroscopy, and elemental microanalysis.

**GC-FID:**

Instrument:	Agilent 6890
Column:	HP-1, 30 m $\times$ 0.32 mm I.D. $\times$ 0.25 $\mu\text{m}$
Program:	150 $^\circ\text{C}$ (2 min), 30 $^\circ\text{C}/\text{min}$ to 290 $^\circ\text{C}$ (16 min), 30 $^\circ\text{C}/\text{min}$ to 300 $^\circ\text{C}$ (5 min)
Injector:	250 $^\circ\text{C}$
Detector Temp:	320 $^\circ\text{C}$
Carrier:	Helium
Split ratio:	20/1

Relative mass fraction of the main component:

Initial analysis:	Mean = 99.8%, s = 0.01% (10 sub samples in duplicate, December 2012)
Re-analysis:	Mean = 99.7%, s = 0.04% (5 sub samples in duplicate, July 2014)
Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, May, 2017)
Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, November, 2021)

**GC-FID:**

Instrument:	Varian CP-3800
Column:	VF-1MS or HP-5, 30 m $\times$ 0.32 mm I.D. $\times$ 0.25 $\mu\text{m}$
Program:	150 $^\circ\text{C}$ (2 min), 30 $^\circ\text{C}/\text{min}$ to 290 $^\circ\text{C}$ (20 min), 30 $^\circ\text{C}/\text{min}$ to 300 $^\circ\text{C}$ (5 min)
Injector:	250 $^\circ\text{C}$
Detector Temp:	320 $^\circ\text{C}$
Carrier:	Helium
Split ratio:	20/1

Relative mass fraction of the main component:

Initial analysis:	Mean = 99.8%, s = 0.02% (10 sub samples in duplicate, December 2012) [VF-1]
Initial analysis:	Mean = 99.8%, s = 0.02% (10 sub samples in duplicate, December 2012) [HP-5]

**Thermogravimetric analysis:** Non volatile residue < 0.2% mass fraction (December 2012). The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material.

**Karl Fischer analysis:** Moisture content < 0.1% mass fraction (December 2012)  
Moisture content 0.12% mass fraction (May 2017)

**qNMR:**

Instrument:	Bruker Avance-400
Field strength:	400 MHz
Solvent:	DMSO- $d_6$ (2.50 ppm)
Internal standard:	1,4-Bis(trimethylsilyl)benzene (100% mass fraction)
Initial analysis:	Mean (4.7 ppm) = 99.5%, s = 0.8% (5 sub samples, March 2013)

**Spectroscopic and other characterisation data**

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 30 m x 0.25 mm I.D. x 0.25 $\mu$ m
	Program:	150 °C (1 min), 10 °C/min to 270 °C (25 min), 30 °C/min to 300 °C (5 min)
	Injector:	250 °C
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	20/1
	The retention time of the parent compound is reported along with the major peaks in the mass spectrum. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (34.7 min):	380 ( $M^+$ -2H, 1), 284 (2), 254 (2), 155 (4), 127 (8), 98 (100), 70 (8), 42 (4) <i>m/z</i>
LC/ESI -MS:	Instrument:	Waters Acquity UPLC/TQD
	Ionisation mode:	Electrospray positive ion
	Capillary voltage:	3.5 kV
	Cone voltage:	60 V
	Source temp:	100 °C
	Desolvation gas temperature:	400 °C
	Cone gas flow rate:	1 L/hr
	Desolvation gas flow rate:	500 L/hr
	Peak:	383.3 ( $M+H^+$ ) <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 $\mu$ 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:	Methylene chloride and hexane
TLC:	Conditions:	Kieselgel 60F <sub>254</sub> . Ethyl acetate /methanol (10/1) Single spot observed, $R_f$ = 0.3. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 $cm^{-1}$ , KBr powder
	Peaks:	3054, 2937, 2853, 2790, 2715, 1607, 1522, 1462, 1395, 1338, 1206, 1184, 1079, 886, 781, 745, 642, 574, 518, 426 $cm^{-1}$
<sup>1</sup> H NMR:	Instrument:	Bruker Avance III-400
	Field strength:	400 MHz
	Solvent:	CDCl <sub>3</sub> (7.26 ppm)
	Spectral data:	$\delta$ 1.04-1.17 (2H, m), 1.23-1.28 (1H, m), 1.41-1.66 (3H, m), 2.12 (1H, dt, $J$ = 3.0, 11.6 Hz), 2.37 (1H, m), 2.41 (3H, s), 2.85 (1H, dm, $J$ = 11.8 Hz), 3.85 (1H, dd, $J$ = 9.2, 13.8 Hz), 4.53 (1H, dd, $J$ = 4.3, 13.7 Hz), 7.34-7.43 (4H, m), 7.45-7.56 (3H, m), 7.67 (1H, dd, $J$ = 1.1, 6.9 Hz), 7.92 (1H, dd, $J$ = 1.3, 8.2 Hz), 7.98 (1H, d, $J$ = 8.2 Hz), 8.21 (1H, d, $J$ = 8.2 Hz), 8.45-8.50 (1H, m) ppm Dichloromethane estimated at 0.4%, ethyl acetate at 0.1% and hexane at 0.1% mass fraction were observed in the <sup>1</sup> H NMR.
<sup>13</sup> C NMR:	Instrument:	Bruker Avance DMX-600
	Field strength:	151 MHz
	Solvent:	CDCl <sub>3</sub> (77.2 ppm)
	Spectral data:	$\delta$ 23.5, 25.6, 29.6, 43.7, 50.0, 57.2, 62.8, 110.3, 117.9, 123.0, 123.1, 123.9, 124.7, 126.17, 126.2, 126.4, 126.9, 127.0, 128.3, 130.3, 131.0, 133.9, 137.6, 138.7, 139.1, 192.2 ppm
Melting point:		134-135 °C
Microanalysis:	Found:	C = 81.4%; H = 6.8%; N = 7.3% (December, 2012)
	Calculated:	C = 81.6%; H = 6.9%; N = 7.3% (Calculated for C <sub>26</sub> H <sub>26</sub> N <sub>2</sub> O)