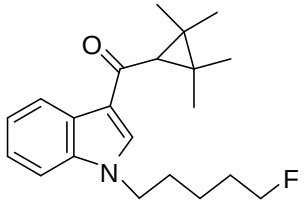
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D1000: 1-(5-Fluoro-pentyl)-3-(2,2,3,3-tetramethylcyclopropoyl)indole

Report ID: D1000.2023.01

Chemical Formula: $C_{21}H_{28}FNO$

Molecular Weight: 329.5 g/mol



Property value

Batch No.	CAS No.	Purity estimate
13-D-17	1364933-54-9	97.8 ± 1.3%

IUPAC name: [1-(5-Fluoropentyl)-1H-indol-3-yl](2,2,3,3-tetramethylcyclopropyl)methanone.

Expiration of certification: The property values are valid till 5 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 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 should be used 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 ºC in a closed container in a dry, dark area.

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.
2 August 2023

This report supersedes any issued prior to 2 August 2023.

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

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The 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 and elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890/8890
	Column:	HP-1/HP-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 20 °C/min to 250 °C (5 min), 30 °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 = 99.2%, s = 0.001% (10 sub samples in duplicate, September 2013)
	Re-analysis:	Mean = 99.2%, s = 0.02% (5 sub samples in duplicate, July 2014)
	Re-analysis:	Mean = 99.2%, s = 0.01% (5 sub samples in duplicate, July 2015)
	Re-analysis:	Mean = 99.2%, s = 0.01% (5 sub samples in duplicate, June 2016)
	Re-analysis:	Mean = 99.1%, s = 0.01% (5 sub samples in duplicate, May 2019)
	Re-analysis:	Mean = 99.2%, s = 0.03% (5 sub samples in duplicate, July 2023)
GC-FID:	Instrument:	Agilent 7890
	Column:	HP-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	60 °C (1 min), 20 °C/min to 250 °C (5 min), 30 °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 = 97.5%, s = 0.03% (10 sub samples in duplicate, September 2013)
Karl Fischer analysis:		Moisture content < 0.2% mass fraction (September 2013, August 2014, July 2015, June 2016, May 2019 and July 2023)
Thermogravimetric analysis:		Volatiles content < 0.1% and non-volatile residue < 0.2% mass fraction (September 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 μm
	Program:	60 °C (1 min), 10 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	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 (22.7 min):	329 (M^+, 62), 314 (70), 270 (40), 256 (30), 247 (40), 232 (100), 144 (64), 130 (27), 116 (26), 41 (24) *m/z*
ESI-MS:	Instrument:	Waters Acquity, UPLC, QBA 119
	Operation:	Positive ion mode, direct infusion at 10 μL/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	EM voltage:	650 V
	Cone voltage:	30 V
	Peak:	330.25 ($M+H^+$) *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, *n*-hexane and methyl cyclopentane
IR:	Instrument:	Biorad FTS300MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3109, 3025, 2977, 2944, 2868, 1618, 1526, 1465, 1413, 1393, 1376, 1225, 1108, 1064, 963, 743 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 1.31 (6H, s), 1.35 (6H, s), 1.50 (2H, m), 1.71 (1H, m), 1.76 (1H, m), 1.95 (3H, m), 4.18 (2H, t, J = 7.1 Hz), 4.44 (2H, dt, J_{H-H} = 5.9 Hz, J_{H-F} = 47.3 Hz), 7.25-7.29 (2H, m), 7.33-7.34 (1H, m), 7.66 (1H, s), 8.42 (1H, m) ppm
	Hexane at 1.4% mass fraction was quantified by ^{1}H NMR.	
^{13}C NMR:	Instrument:	Bruker Avance III-600
	Field strength:	150 MHz
	Solvent:	$CDCl_3$ (77.16 ppm)
	Spectral data:	δ 17.2, 23.0 (d, J_{C-F} = 4.8 Hz), 24.2, 29.8, 30.1 (d, J_{C-F} = 20 Hz), 31.7, 41.8, 47.0, 83.8 (d, J_{C-F} = 165.1 Hz), 109.7, 119.9, 122.3, 122.9, 123.1, 126.5, 133.5, 136.7, 194.8 ppm
^{19}F NMR:	Instrument:	Bruker Avance-400
	Field strength:	376 MHz
	Solvent:	$CDCl_3$
	Spectral data:	δ -218.7 ppm
Melting point:		69-71 °C
Microanalysis:	Found:	C = 76.5%; H = 8.8%; N = 4.2%; F = 5.6% (September 2013)
	Calculated:	C = 76.6%; H = 8.6%; N = 4.3%; F = 5.8% (for $C_{21}H_{28}FNO$)