



### REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D1000.2016.01

Compound Name: 1-(5-Fluoro-pentyl)-3-(2,2,3,3-tetramethylcyclopropyl)indole

Description: White powder

Collection Number: D1000

Chemical Formula: C<sub>21</sub>H<sub>28</sub>FNO

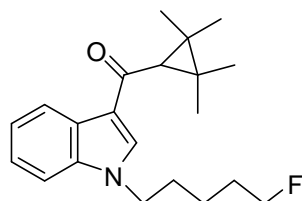
CAS Number: 1364933-54-9

Structure:

Batch Number: 13-D-17

Molecular Weight: 329.5

Release date: 3<sup>rd</sup> October 2013



Synonyms: XLR-11

5-Fluoro-UR-144

5-F-UR-144

(1-(5-Fluoropentyl)-1H-indol-3-yl)(2,2,3,3-tetramethylcyclopropyl)-methanone

Purity (mass fraction): 94.5 ± 3.7% (95% coverage interval)

The purity estimate was obtained by traditional analytical techniques by subtraction from 100% of total impurities by GC-FID, thermogravimetric analysis, Karl Fischer analysis and <sup>1</sup>H NMR. Supporting evidence is provided by headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID: Instrument: Agilent 6890  
Column: HP-1, 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 response of main component:  
Initial analysis: Mean = 97.7%, s = 0.04% (10 sub samples in duplicate, September 2013)  
Re-analysis: Mean = 98.3%, s = 0.05% (5 sub samples in duplicate, July 2014)  
Re-analysis: Mean = 98.2%, s = 0.01% (5 sub samples in duplicate, July 2015)  
Re-analysis: Mean = 98.1%, s = 0.02% (5 sub samples in duplicate, June 2016)

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 response of main component:  
Initial analysis: Mean = 97.5%, s = 0.03% (10 sub samples in duplicate, September 2013)

Thermogravimetric analysis: Non volatile residue < 0.2% mass fraction (September 2013). 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.

Karl Fischer analysis: Moisture content < 0.2% mass fraction (September 2013, August 2014, July 2015, and June 2016)

### 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:	60 $^{\circ}$ C (1 min), 10 $^{\circ}$ C/min to 300 $^{\circ}$ C (3 min)
	Injector:	250 $^{\circ}$ C
	Carrier:	Helium, 1.0 mL/min
		Transfer line temp: 280 $^{\circ}$ C
		Split ratio: 20/1
	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 (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 $\mu$ L/min
	Ionisation:	ESI spray voltage at 3.5 kV positive ion
	Cone voltage:	30 V
	Source temp:	100 $^{\circ}$ C
		Desolvation gas temperature: 200 $^{\circ}$ C
	Cone gas flow rate:	2 L/hr
		Desolvation gas flow rate: 500 L/hr
	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 $\mu$ m
	Program:	50 $^{\circ}$ C (5 min), 7 $^{\circ}$ C/min to 120 $^{\circ}$ C, 15 $^{\circ}$ C/min to 220 $^{\circ}$ C (8.3 min)
	Injector:	150 $^{\circ}$ C
		Transfer line temp: 280 $^{\circ}$ 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 $cm^{-1}$
$^1H$ NMR:	Instrument:	Bruker Avance-600
	Field strength:	600 MHz
		Solvent: $CDCl_3$ (7.26 ppm)
	Spectral data:	$\delta$ 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 Dichloromethane (0.5%) and n-hexane (2.7%) estimated mass fraction was observed in the $^1H$ NMR.
$^{13}C$ NMR:	Instrument:	Bruker Avance-600
	Field strength:	150 MHz
		Solvent: $CDCl_3$ (77.16 ppm)
	Spectral data:	$\delta$ 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:	$\delta$ -218.7 ppm.
Melting point:	69-71 $^{\circ}$ C	
Microanalysis:	Found: C = 76.5%; H = 8.8%; N = 4.2%; F = 5.6% (September 2013) Calc: C = 76.6%; H = 8.6%; N = 4.3%; F = 5.8% (for $C_{21}H_{28}FNO$ )	

Accredited for compliance with ISO Guide 34.

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### Expiration of certification

The property values are valid till 3<sup>rd</sup> June 2019, i.e. three 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.

The long-term stability of the compound in solution has not been examined.

This material has demonstrated stability over a minimum period of three years. The measurement uncertainty at the 95% coverage interval includes a stability component which has been estimated from annual stability trials.

### 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.

### 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.

### Intended use

For *in vitro* laboratory analysis only.

### Caution

Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

### Legal notice

Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R Davies  
Team Leader,  
Chemical Reference Materials, NMI  
Dated: 15 June 2016

Characterisation data and property values specified in this report supersede all reports issued prior to 15<sup>th</sup> June 2016.