Australian Government
Department of Industry, Innovation and Science

National Measurement Institute

NATA
ACCREDITED FOR TECHNICAL COMPETENCE

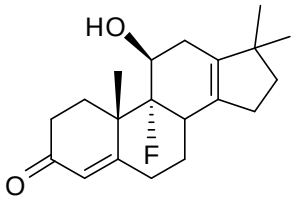
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D571: 9α-Fluoro-17,17-dimethyl-18-norandrosta-4,13-diene-11β-ol-3-one

Report ID: D571.2021.01 (Ampouled 110815)

Chemical Formula: $C_{20}H_{27}FO_2$

Molecular Weight: 318.4 g/mol



Certified value

Batch No.	CAS No.	Mass per ampoule
99-S-02	3863-16-9	1001 ± 14 µg

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

IUPAC name: (11β)-9-fluoro-11-hydroxy-10,17,17-trimethylgona-4,13-dien-3-one.

Expiration of certification: The property values are valid till 22 February 2031, i.e. ten 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 ampoules that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials.

Description: The compound is supplied as a dried aliquot in a sealed ampoule under an atmosphere of argon. The CRM is intended for a single use to prepare a standard solution containing D571. Material was sourced from an external supplier and certified for identity and purity by NMIA.

Intended use: This certified reference material is suitable for use as a primary calibrator.

Instructions for use: Open the ampoule and carefully rinse the interior at least three times with a suitable organic solvent (e.g. chloroform). This will transfer 1001 ± 14 µg of anhydrous 9α-fluoro-17,17-dimethyl-18-norandrosta-4,13-diene-11β-ol-3-one. The mass of analyte in each ampoule is calculated from the assigned purity of the bulk and the concentration of bulk material in a stock solution used to prepare the ampoules.

Recommended storage: When not in use, this material should be stored at or below 4 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 approach all impurities are quantified as a mass fraction and subtracted from 100%.

Stability: This material has demonstrated stability over a minimum period of ten 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 seven randomly selected ampoules 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 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 March 2021

This report supersedes any issued prior to 02 March 2021

Characterisation Report:

Warning: This material is sensitive to the quality of the silanised glass liner when injected at elevated temperature (~ 250 ºC) into a GC instrument.

GC-FID:	Instrument:	Agilent 6890 or 7890	
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm	
	Program:	180 °C (1 min), 20 °C/min to 260 °C, 10 °C/min to 300 °C (3 min)	
	Injector:	250 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative mass fraction of the main component:		
	Initial analysis:	Mean = 99.6%, s = 0.002% (7 ampoules in duplicate, August 2011)	
	Re-analysis:	Mean = 99.4%, s = 0.4% (5 ampoules in duplicate, August 2012)	
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 ampoules in duplicate, July 2015)	
	Re-analysis:	Mean = 99.6%, s = 0.01% (5 ampoules in duplicate, May 2018)	
	Re-analysis:	Mean = 99.4%, s = 0.06% (5 ampoules in duplicate, February 2021)	

The following analytical data was obtained on the bulk material subsequently used in the preparation of the ampoules.

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 elemental microanalysis.

Warning: This material is sensitive to the quality of the silanised glass liner when injected at elevated temperature (~ 250 ºC) into a GC instrument.

GC-FID:	Instrument:	HP 5890	
	Column:	ZB-1, 30 m × 0.32 mm I.D. × 0.25 μm	
	Program:	180 °C (1 min), 10 °C/min to 220 °C, 20 °C/min to 300 °C (6 min)	
	Injector:	250 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative mass fraction of main component:		
	Initial analysis:	Mean = 99.0%, s = 0.09% (3 sub samples in duplicate, April 2003)	
	Re-analysis:	Mean = 99.7%, s = 0.01% (2 sub samples in duplicate, May 2004)	
	Re-analysis:	Mean = 99.4%, s = 0.01% (5 sub samples in duplicate, June 2005)	
GC-FID:	Instrument:	Agilent 6890	
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm	
	Program:	180 °C (1 min), 15 °C/min to 300 °C (6 min)	
	Injector:	250 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative mass fraction of main component:		
	Initial analysis:	Mean = 99.5%, s = 0.02% (5 sub samples in duplicate, May 2008)	
GC-FID:	Instrument:	Agilent 6890	
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 μm	
	Program:	180 °C (1 min), 20 °C/min to 260 °C, 10 °C/min to 300 °C (3 min)	
	Injector:	250 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative peak area of main component:		
	Initial analysis:	Mean = 99.6%, s = 0.01% (7 sub samples in duplicate, August 2011)	
Thermogravimetric analysis:		Volatile content < 0.1% and non volatile residue < 0.2% mass fraction (November 1999 and June 2005)	
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (May 2008)	

Spectroscopic and other characterisation data

GC-MS:

Parent compound:

Instrument:	Agilent 6890/5973
Column:	HP Ultra 2, 17 m x 0.22 mm I.D. x 0.11 μm
Program:	140 °C (1 min), 8 °C/min to 250 °C, 30 °C/min to 300 °C (3 min)
Injector:	280 °C Transfer line temp: 300 °C
Carrier:	Helium, 1.0 mL/min Split ratio: 30/1
Scan range:	50-550 m/z

Bis-TMS derivative:

Column:	HP Ultra 1, 17 m × 0.22 mm I.D. × 0.11 μm
Program:	170 °C, 3 °C/min to 234 °C, 10 °C/min to 265 °C (3min)
Injector:	280 °C Transfer line temp: 300 °C
Carrier:	Helium, 1.0 mL/min Split ratio: 30/1
Scan range:	50-550 m/z

The retention times of the parent compound and *bis*-TMS derivative are reported along 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 (9.1 min): 318 (M^+, 16), 303 (100), 285 (15), 270 (9), 265 (16), 243 (10) *m/z*
Bis-TMS (10.8 min): 462 (M^+, 60), 447 (3), 357 (8), 337 (6), 208 (47), 73 (100) *m/z*

The *bis*-silylated derivative of the synthetic material co-elutes with a derivatised comparison sample and the two materials produce matching mass spectra.

TLC:

Conditions:	Kieselgel 60F_{254} Chloroform/ethyl acetate (5:1) Single spot observed, R_f = 0.30 (5 sub samples)

IR:

Instrument:	FT-IR, Biorad WIN FTS40
Range:	4000-400 cm^{-1}, KBr powder
Key peaks:	3342, 1651, 1454, 1358, 1039, 877 cm^{-1}

^{1}H NMR:

Instrument:	Bruker Advance-300
Field strength:	300 MHz
Solvent:	$CDCl_3$ (7.26 ppm)
Key spectral data:	δ 0.95 (3H, s), 0.97 (3H, s), 1.48 (3H, s), 4.29 (1H, br s), 5.79 (1H, d, *J* = 1.6 Hz) ppm Ethyl acetate estimated at 0.5% mass fraction was observed in the 1H NMR (March 1999)

^{13}C NMR:

Instrument:	Bruker Advance-300
Field strength:	75 MHz
Solvent:	$CDCl_3$ (77.0 ppm)
Spectral data:	δ 20.5 (d), 24.0 (d), 26.2, 26.5, 28.6 (d), 28.8 (d), 30.0, 31.7, 33.5, 40.0 (d), 39.0, 43.1 (d), 45.4, 65.9 (d), 97.9 (d), 125.7, 131.5, 137.6, 168.6, 198.7 ppm

Melting point: 212-213 °C

Microanalysis:

Found:	C = 75.6%; H = 8.6%; F = 6.1% (April, 1999)
Calculated:	C = 75.4%; H = 8.6%; F = 6.0% (Calculated for $C_{20}H_{27}FO_2$)