Australian Government
Department of Industry, Science and Resources
National Measurement Institute
NATA WORLD RECOGNISED ACCREDITATION

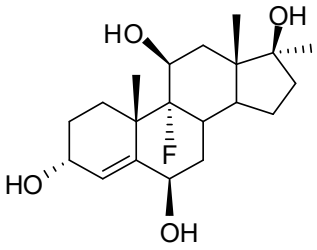
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D616: 9α-Fluoro-17α-methyl-4-androsten-3α, 6β, 11β, 17β-tetrol

Report ID: D616.2026.01 (Ampouled 220818)

Chemical Formula: $C_{20}H_{31}FO_4$

Molecular Weight: 354.5 g/mol



Property value

Batch No.	CAS No.	Mass per ampoule
99-S-24	148505-57-1	990 ± 24 µg

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

IUPAC name: (3α,6β,11β,17β)-9-Fluoro-17-methylandrost-4-ene-3,6,11,17-tetrol

Expiration of certification: The property values are valid till 28 April 2036, 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 reference material is intended for a single use to prepare a standard solution containing D616. The material was sourced from an external supplier and certified for identity and purity by NMI Australia.

Intended use: This reference material is recommended for qualitative analysis only and is not intended for use as a calibrator. The material does not have certified reference material status as metrological traceability of the stated purity value to the SI unit for mass (kg) has not been established.

Instructions for use: Open the ampoule and carefully rinse the interior at least three times, each time with a minimum of 0.5 mL of a suitable organic solvent (e.g. methanol). This process will ensure the transfer of the stated mass per ampoule of anhydrous 9α-fluoro-17α-methyl-4-androsten-3α, 6β, 11β, 17β-tetrol. 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.

Stability: In the absence of long term stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated from stability trials conducted on similar materials by NMI Australia over the last ten years. The measurement uncertainty at the 95% confidence interval also includes a stability component determined from accelerated stability trials conducted at 40 ºC and 75% humidity for 14 days.

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 UHPLC with UV detection 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.
29 April 2026

This report supersedes any issued prior to 29 April 2026.

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.

Characterisation Report:

HPLC:	Instrument:	Thermo Scientific Vanquish Flex
	Column:	ACE C-18, 5 μm (4.6 mm x 250 mm)
	Column oven:	40 °C
	Mobile Phase:	Acetonitrile/Milli Q water A = Milli Q water; B = Acetonitrile 0-12 min 16% B; 12-20 min 16-80% B; 20-23 min 80% B; 23-24 min 80-16% B; 24-40 min 16% B.
	Flow rate:	0.8 mL/min
	Detector:	Vanquish PDA operating at 201 nm
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.0%, s = 0.01% (7 ampoules in duplicate, August 2022)
	Re-analysis:	Mean = 97.4%, s = 0.1% (5 ampoules in duplicate, June 2023)
	Re-analysis:	Mean = 98.5%, s = 0.1% (5 ampoules in duplicate, June 2024)
	Re-analysis:	Mean = 98.3%, s = 0.03% (5 ampoules in duplicate, May 2025)
	Re-analysis:	Mean = 98.8%, s = 0.01% (5 ampoules in duplicate, April 2026)

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

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 UHPLC-UV detection, thermogravimetric analysis, Karl Fischer analysis, and ^{1}H NMR spectroscopy. The purity value is calculated as per Equation 1.

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

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

Supporting evidence is provided by elemental microanalysis.

HPLC:	Instrument:	Thermo Scientific UltiMate 3000
	Column:	ACE C-18, 5 μm (4.6 mm x 250 mm)
	Column oven:	40 °C
	Mobile Phase:	Acetonitrile/Milli Q water A = Milli Q water; B = Acetonitrile 0-18 min 20% B; 18-23 min 20-80% B; 23-28 min 80%B; 28-29 min 80-20%B; 29-35min 20%B.
	Flow rate:	0.8 mL/min
	Detector:	RS Diode Array at 201 nm
	Relative peak area of the main component:	
	Initial analysis:	Mean = 98.9%, s = 0.03% (7 sub samples in duplicate, August 2022)
Karl Fischer analysis:		Moisture content 0.1% mass fraction (August 2022)
Thermogravimetric analysis:		Volatiles content 0.3% and non-volatile residue < 0.2% mass fraction (November 1999 & October 2005)

Spectroscopic and other characterisation data

GC-MS:	*Tetra*-TMS derivative:	
	Instrument:	HP 6890/5973
	Column:	HP Ultra 2, 17 m x 0.22 mm I.D. x 0.11 μm
	Program:	170 °C (0.5 min), 3 °C/min to 234 °C, 10 °C/min to 265 °C (3 min)
	Injector:	280 °C
	Split ratio:	20/1
	Transfer line temp:	300 °C
	Carrier:	Helium
	Scan range:	50-550 *m/z*
	The retention time of *tetra*-TMS derivative 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.	
	Tetra-TMS (13.5 min):	642 (M^+, 2), 552 (7), 462 (4), 357 (3), 143 (86), 73 (80) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Ethyl acetate Single spot observed, R_f = 0.3
IR:	Instrument:	Perkin-Elmer FT-IR
	Range:	4000-400 cm^{-1}, Nujol mull
	Peaks:	3592, 3328, 2357, 1454, 1377, 1266, 994, 934 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Spectral data:	δ 1.00 (3H, s), 1.05, 1.42 (3H, s), 1.51 (3H, s), 3.89 (1H, br s), 3.99 (1H, br s), 4.1 (1H, br d), 5.50 (1H, d) ppm
		Signals consistent with the 3β-hydroxy isomer are observed in the ^{1}H NMR spectrum at a relative mass fraction of 1%.
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	DMSO-d_6 (39.52 ppm)
	Spectral data:	δ 16.3, 23.4, 25.2, 25.4, 26.7, 27.9, 31.2 (d), 34.4, 37.0, 38.5, 42.1 (d), 44.3, 45.2, 62.2, 69.7 (d), 71.8, 80.4, 101.0 (d), 127.5, 146.5 ppm
Melting point:		201-203 °C
Microanalysis:	Found:	C = 67.7%, H = 8.8%
	Calculated:	C = 67.8%, H = 8.8% (Calculated for $C_{20}H_{31}FO_4$)