Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

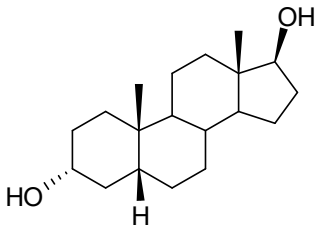
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D636: 5β-Androstane-3α,17β-diol

Report ID: D636.2025.02 (Ampouled 181108)

Chemical Formula: $C_{19}H_{32}O_2$

Molecular Weight: 292.46 g/mol



Certified value

Batch No.	CAS No.	Mass per ampoule
98-001017	**1851-23-6**	**1000 ± 18 µg**

The uncertainty has been calculated according to ISO 33405 and is stated at the 95% confidence limit (k = 2).

IUPAC: (3α,5β,17β)-Androstane-3,17-diol

Expiration of certification: The property values are valid till 23 July 2032, seven 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. The material will be re-tested periodically to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

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 D636. This material was sourced from an external supplier and certified for identity and purity by NMI Australia.

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, 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 5β-androstane-3α, 17β-diol. The mass of analyte contained in each ampoule has been determined based on the assigned purity of the bulk material and the concentration of that bulk material in the stock solution used during ampoule preparation.

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 three 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.
15 July 2026

This report supersedes any issued prior to 15 July 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:

GC-FID:

Instrument:	Varian CP-3800
Column:	HP-1 or HP-5, 30 m x 0.32 mm I.D. x 0.25 μm
Program:	230 °C (0.2 min), 5 °C/min to 265 °C, 30 °C/min to 280 °C (3 min)
Injector:	250 °C
Detector Temp:	320 °C
Carrier:	Helium
Split ratio:	20/1

Relative mass fraction of the main component as the *bis*-TMS derivative:

Initial analysis:	Mean = 99.8%, s = 0.01% (7 ampoules in duplicate, December 2018)
Re-analysis:	Mean = 99.9%, s = 0.01% (5 ampoules in duplicate, September 2021)
Re-analysis:	Mean = 99.7%, s = 0.06% (4 ampoules in duplicate, July 2025)

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 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.

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

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

Supporting evidence is provided by elemental microanalysis.

GC-FID:

Instrument:	Agilent 6890N or Varian CP-3800
Column:	30 m x 0.32 mm I.D. x 0.25 μm
Program:	215 °C (20 min), 20 °C/min to 300 °C (5 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% (10 sub samples in duplicate, 1999)
Re-analysis:	Mean = 99.8%, s = 0.1% (10 sub samples in duplicate, February 2005) (HP-1)
Re-analysis:	Mean = 99.6%, s = 0.1% (10 sub samples in duplicate, February 2010) (HP-1)
Initial analysis:	Mean = 99.8%, s = 0.1% (10 sub samples in duplicate, February 2010) (HP-5)
Initial analysis:	Mean = 99.5%, s = 0.1% (10 sub samples in duplicate, February 2010) (VF-1)
Initial analysis:	Mean = 99.9%, s = 0.04% (10 sub samples in duplicate, February 2010) (VF-1)
Initial analysis:	Mean = 99.8%, s = 0.1% (10 sub samples in duplicate, February 2010) (HP-1)

GC-FID:

Instrument:	Varian CP-3800
Column:	HP-1, 30 m x 0.32 mm I.D. x 0.25 μm
Program:	230 °C (0.2 min), 5 °C/min to 265 °C (2 min), 30 °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 as the *bis*-TMS derivative:

Initial analysis:	Mean = 99.9%, s = 0.01% (10 sub samples in duplicate, April 2010)
Re-analysis:	Mean = 99.9%, s = 0.02% (5 sub samples in duplicate, April 2015)

GC-FID:

Instrument:	Agilent 5890
Column:	ZB-1, 30 m x 0.32 mm I.D. x 0.25 μm
Program:	230 °C (0.2 min), 5 °C/min to 265 °C (2 min), 30 °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 as the *bis*-TMS derivative:

Initial analysis:	Mean = 99.7%, s = 0.03% (10 sub samples in duplicate, April 2010)

Thermogravimetric analysis: The volatile content (e.g. organic solvents and/or water) could not be analysed accurately because of the inherent volatility of the material. Non volatile residue < 0.2 % mass fraction (February 2010)

Karl Fischer analysis: Moisture content < 0.2% mass fraction (February 2010)
Moisture content < 0.1% mass fraction (April 2015)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Columns:	ZB-5 MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	100 °C, 15 °C/min to 230 °C, then 8 °C/min to 310 °C
	Injector:	250 °C
	Transfer line temp:	300 °C
	Split ratio:	20/1
	The retention time of the parent material 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.	
	9.4 min:	292 (M^+, 6), 274 (65), 256 (52), 241 (47), 215 (100) *m/z*
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	3343, 2924, 2864, 1469, 1452, 1367, 1276, 1172, 1056, 1036 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$/DMSO-d_6 (7.26 ppm/2.5 ppm)
	Key spectral data:	δ 0.55 (3H, s), 0.76 (3H, s), 3.42 (1H, m), 3.44 (1H, t) ppm
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	126 MHz
	Solvent:	MeOH-d_4/DMSO-d_6 (49.0 ppm/39.52 ppm)
	Spectral data:	δ 11.1, 20.3, 23.2, 23.3, 26.1, 27.1, 30.0, 30.4, 34.6, 35.5, 36.0, 36.4, 37.2, 40.8, 42.2, 43.0, 51.2, 70.5, 80.7 ppm
Microanalysis:	Found:	C = 78.1%; H = 11.1% (May 2000)
	Calculated:	C = 78.0%; H = 11.0% (Calculated for $C_{19}H_{32}O_2$)