Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

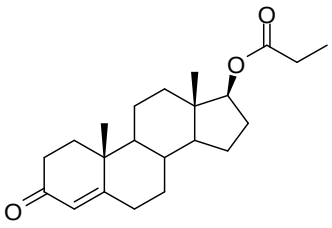
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D710b: Testosterone propionate

Report ID: D710b.2024.01 (Bottled 240305)

Chemical Formula: $C_{22}H_{32}O_3$

Molecular Weight: 344.5 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
21-S-01	57-85-2	98.9 ± 0.9%

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

IUPAC name: (17β)-3-Oxoandrost-4-en-17-yl propionate.

Expiration of certification: The property values are valid till 17 April 2027, 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 material will be re-tested on an annual basis 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: Off-white powder prepared by synthesis and certified for identity and purity by NMIA. Packaged in amber glass bottles with a septum and crimped aluminium cap.

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

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 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 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 and accelerated stability trials, the latter conducted at 40 ºC and 75% humidity for a 14 day period.

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.
23 April 2024

This report supersedes any issued prior to 23 April 2024

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:

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:	Agilent 8890	
	Column:	HP-1MS, 30 m x 0.32 mm I.D. x 0.25 μm	
	Program:	220 °C (1 min), 10 °C /min to 250 °C (1 min), 5 °C /min to 280 °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 mass fraction of the main component:		
	Initial analysis:	Mean = 99.5%, s = 0.02% (10 sub samples in duplicate, June 2021)	
	Re-analysis:	Mean = 99.5%, s = 0.01% (10 sub samples in duplicate, April 2024)	
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (June 2021) Moisture content < 0.1% mass fraction (April 2024)	
Thermogravimetric analysis:		Volatile content 0.1% and non volatile residue < 0.2% mass fraction (June 2021)	

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Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	DB-5MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	220 °C (1 min), 15 °C /min to 280 °C (5 min), 30 °C /min to 300 °C (1 min)
	Injector:	250 °C Transfer line temp: 280 °C
	Carrier:	Helium, 1.0 mL/min Split ratio: 30/1
	The retention time of the parent compound 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.	
	Parent (9.4 min):	344 (M^+, 20), 302 (15), 288 (5), 228 (27), 185 (16), 148 (16), 147 (69), 146 (23), 133 (19), 124 (77), 105 (26), 91 (34), 57 (100) *m/z*
TLC:	Conditions:	Kieselgel 60F254. Hexane/ethyl acetate (7/3) Single spot observed, Rf = 0.3. Visualisation with UV at 254 nm
IR:	Instrument:	Bruker Alpha FT-IR
	Range:	4000-400 cm^{-1}, neat Peaks: 2968, 2911, 2851, 2826, 1723, 1666, 1610, 1434, 1388, 1332, 1237, 1077, 1042, 1019, 861 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance-III
	Field strength:	500 MHz
	Solvent:	Benzene-d_6 (7.16 ppm)
	Spectral data:	δ 0.46 (1H, m), 0.55-0.65 (2H, m), 0.69 (3H, s), 0.74 (3H, m), 1.01 (3H, t, *J* = 7.5 Hz), 0.97-1.17 (5H, m), 1.23 (1H, ddd, *J* = 4.7, 14.4, 14.4 Hz), 1.28-1.38 (2H, m), 1.42-1.53 (2H, m), 1.73 (1H, m), 1.80-1.91 (2H, m), 2.07-2.20 (2H, m), 2.12 (2H, q, *J* = 7.5 Hz), 2.26 (1H, ddd, *J* = 4.0, 4.0, 16.6 Hz), 4.74 (1H, dd, *J* = 8.4, 8.9 Hz), 5.83 (1H, s) ppm Hexane estimated at 0.6% mass fraction was observed in the ^{1}H NMR.
^{13}C NMR:	Instrument:	Bruker Avance-III
	Field strength:	125 MHz
	Solvent:	Benzene-d_6 (128.1 ppm)
	Spectral data:	δ 9.5, 12.3, 17.1, 20.7, 23.6, 27.9, 28.0, 31.6, 32.6, 34.3, 35.4, 35.9, 37.0, 38.4, 42.7, 50.2, 53.7, 82.3, 124.6, 168.5, 173.7, 197.2 ppm
Melting point:		120-122 °C
Microanalysis:	Found:	C = 76.8%; H = 9.4% (June 2021)
	Calculated:	C = 76.7%; H = 9.4% (Calculated for $C_{22}H_{32}O_3$)