Australian Government
Department of Industry, Innovation and Science

National Measurement Institute

NATA ACCREDITED FOR TECHNICAL COMPETENCE

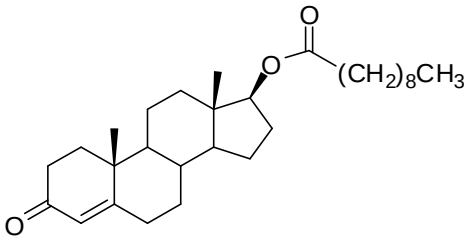
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D683: Testosterone decanoate

Report ID: D683.2018.02 (Bottled 180409)

Chemical Formula: $C_{29}H_{46}O_3$

Molecular Weight: 442.7 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
01-S-06	5721-91-5	99.9 ± 0.3%

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 decanoate

Expiration of certification: The property values are valid till 15 May 2023, 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.

Description: 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 or screw top 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 25 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%. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

Stability: This material has demonstrated stability over a minimum period of five 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 HPLC with UV detection on five 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.
1 April 2020

This report supersedes any issued prior to 1 April 2020

NATA logo notice: Accredited for compliance with ISO 17034. Accreditation No. 198 / Corporate Site No. 20844. The results of the tests, calibrations and/or measurements included in this document are traceable to Australian/national standards.

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 HPLC with UV/ELS detection, 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 qualitative elemental microanalysis.

HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Altima C-18, 5 µm (4.6 mm × 150 mm)
	Column oven:	Ambient
	Mobile Phase:	Methanol/MilliQ water (95:5 v/v)
	Flow rate:	1.0 mL/min
	Detector:	Waters 2998 PDA operating at 243 nm Waters ELSD 2424
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.8%, s = 0.1% (10 sub samples in duplicate, April 2001)
	Re-analysis:	Mean = 99.9%, s =0.02% (5 sub samples in duplicate by ELSD, April 2008) Mean = 99.9%, s = 0.004% (5 sub samples in duplicate by UV April 2008)
	Re-analysis:	Mean = 99.9%, s =0.02% (5 sub samples in duplicate by ELSD, April 2013) Mean = 99.9%, s = 0.01% (5 sub samples in duplicate by UV April 2013)
HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	Altima C-18, 5 µm (4.6 mm × 150 mm)
	Column oven:	Ambient
	Mobile Phase:	Acetonitrile/MilliQ water (95:5 v/v)
	Flow rate:	1.0 mL/min
	Detector:	Waters 2998 PDA operating at 243 nm Waters ELSD 2424
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.7%, s = 0.04% (7 sub samples in duplicate by UV, May 2018) Mean = 99.9%, s = 0.07% (7 sub samples in duplicate by ELSD, May 2018)
Karl Fischer analysis:		Moisture content ≤ 0.10% mass fraction (March 2008 and February 2013) Moisture content ca 0.13% mass fraction (March 2018)
Thermogravimetric analysis:		Volatile content < 0.1% and non volatile residue < 0.2 % mass fraction (April 2001, April 2005 and April 2008)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP6890
	Column:	Zebron ZB-5, 5% phenyl siloxane, 30 m × 0.25 mm I.D. × 0.30 µm
	Program:	180 °C (1.0 min), 10 °C /min to 260 °C, 20 °C /min to 300 °C (3 min)
	Injector:	260 °C
	Split ratio:	20/1
	Transfer line temp:	300 °C
	Carrier:	Helium, 1mL/min
	Scan range:	50-550 *m/z*
	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 (7.30 min):	442 (M^+, 24), 400 (14), 288 (24), 271 (25), 230 (32), 228 (33), 147 (86), 124 (100) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/ethyl acetate (4:1) Single spot observed, R_f = 0.28 (3 samples)
IR:	Instrument:	Perkin-Elmer FT-IR
	Range:	4000-400 cm^{-1}, KBr disc
	Peaks:	2916, 2850, 1735, 1670, 1471, 1328, 1255, 1216, 1181 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Key spectral data:	δ 0.83 (3H, s), 0.87 (3H, t), 1.18 (3H, s), 4.60 (1H, dd), 5.71 (1H, s) ppm
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	126 MHz
	Solvent:	$CDCl_3$ (77.2 ppm)
	Spectral data:	δ 11.9, 14.0, 17.3, 20.4, 22.5, 23.4, 25.0, 27.4, 29.0, 29.1, 29.3, 31.4, 31.7, 32.6, 33.8, 34.5, 35.3, 35.6, 36.5, 38.5, 42.4, 50.2, 53.6, 82.0, 123.9, 170.8, 173.7, 199.2 ppm
Melting point:		48-49.5 °C
Microanalysis:	Found:	C = 78.7%; H = 10.6% (June 2001)
	Calculated:	C = 78.7%; H = 10.5% (Calculated for $C_{29}H_{46}O_3$)