Australian Government
Department of Industry, Innovation and Science

National Measurement Institute

NATA ACCREDITED FOR TECHNICAL COMPETENCE

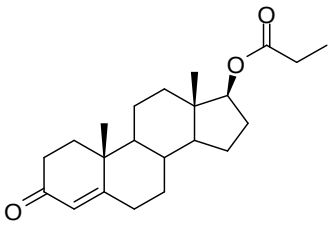
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D710: Testosterone propionate

Report ID: D710.2017.02

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

Molecular Weight: 344.5 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
15-S-02	**57-85-2**	**99.7 ± 1.3%**

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 propionate.

Expiration of certification: The property values are valid till 17 May 2022, i.e. five 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: Off-white powder sourced from an external supplier, 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 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%. 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 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 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.
27 August 2019

This report supersedes any issued prior to 27 August 2019

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 quantitative nuclear magnetic resonance (QNMR) and 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 6890N or Agilent 7890A	
	Column:	HP-1, 30 m x 0.32 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 (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 = 99.8%, s = 0.004% (10 sub samples in duplicate, May 2015)	
	Re-analysis:	Mean = 99.7%, s = 0.01% (5 sub samples in duplicate, April 2016)	
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, May 2017)	
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (May 2015) Moisture content < 0.1% mass fraction (April 2016) Moisture content 0.1% mass fraction (April 2017)	
Thermogravimetric analysis:		Volatile content 0.1% and non volatile residue < 0.2% mass fraction (May 2015)	
QNMR:	Instrument:	Bruker Avance-III-500	
	Field strength:	500 MHz	Solvent: DMSO-d_6 (2.50 ppm)
	Internal standard:	Dimethyl terephthalate (100% mass fraction)	
	Initial analysis:	Mean (5.62 ppm) = 100.1%, s = 0.4% (5 sub samples, May 2015)	
	Initial analysis:	Mean (4.49 ppm) = 99.8%, s = 0.3% (5 sub samples, May 2015)	

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	TG-1MS, 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 (8.10 min):	344 (M^+, 32), 302 (24), 288 (10), 228 (37), 185 (23), 148(21), 147 (86), 146 (30), 133 (22), 124 (100), 105 (26), 91 (33), 57 (94) *m/z*
TLC:	Conditions:	Kieselgel 60F254. Hexane/ethyl acetate (7/3) Single spot observed, Rf = 0.26. Visualisation with UV at 254 nm
IR:	Instrument:	Bruker Alpha FT-IR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	2938, 2911, 2851, 1723, 1666, 1610, 1184, 1131, 861 cm^{-1}
1H NMR:	Instrument:	Bruker Avance-III
	Field strength:	500 MHz Solvent: Benzene-d_6 (7.16 ppm)
	Spectral data:	δ 0.46 (1H, m), 0.54-0.66 (2H, m), 0.69 (3H, s), 0.75 (3H, m), 1.02 (3H, t, J = 7.5 Hz), 0.96-1.18 (5H, m), 1.22 (1H, ddd, *J* = 4.7, 14.4, 14.4 Hz), 1.27-1.39 (2H, m), 1.44 (1H, ddd, *J* = 3.0, 4.3, 13.4 Hz), 1.51 (1H, m), 1.74 (1H, dd, *J* = 2.2, 8.4 Hz), 1.84 (1H, m), 1.87 (1H, ddd, *J* = 4.9, 14.4. 19.4 Hz), 2.08-2.23 (2H, m), 2.12 (2H, q, *J* = 7.5 Hz), 2.27 (1H, ddd, *J* = 4.0, 4.0, 16.6 Hz), 4.74 (1H, dd, *J* = 8.3, 8.3 Hz), 5.83 (1H, s) ppm Hexane estimated at 0.1% mass fraction was observed in the ^{1}H NMR.
13C 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, 53.6, 82.3, 124.6, 168.5, 173.7, 197.2 ppm
Melting point:		122-123 °C
Microanalysis:	Found:	C = 76.8%; H = 9.4% (May 2015)
	Calculated:	C = 76.7%; H = 9.4% (Calculated for $C_{22}H_{32}O_3$)