Australian Government
Department of Industry, Science and Resources

National Measurement Institute

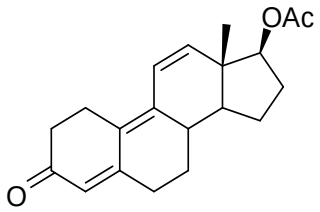
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA S024: Trenbolone acetate

Report ID: S024.2021.03 (Bottled 160215)

Chemical Formula: $C_{20}H_{24}O_3$

Molecular Weight: 312.4 g/mol



Property value

Batch No.	CAS No.	Purity estimate
14-S-01	10161-34-9	95.3 ± 1.5 %

IUPAC name: (17β)-3-Oxoestra-4,9,11-trien-17-yl acetate.

Expiration of certification: The property values are valid till 31 March 2026, 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: Light yellow solid 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 reference material is recommended for qualitative analysis only.

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.

Stability: In the absence of long term stability data the stability of this material has been judged from stability trials conducted on similar materials by NMI Australia over the last ten years. 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.
17 November 2022

This report supersedes any issued prior to 17 November 2022.

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 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 qualitative elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800 or Agilent 7890
	Column:	HP-5 or HP-1, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	180 °C (1 min), 30 °C/min to 250 °C (10 min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 98.6%, s = 0.06% (10 sub samples in duplicate, February 2014)
	Re-analysis:	Mean = 98.4%, s = 0.02% (5 sub samples in duplicate, February 2015)
	Re-analysis:	Mean = 98.4%, s = 0.02% (5 sub samples in duplicate, February 2015)
	Re-analysis:	Mean = 95.9%, s = 0.08% (5 sub samples in duplicate, March 2017)
	Re-analysis:	Mean = 95.2%, s = 0.10% (5 sub samples in duplicate, March 2018)
	Re-analysis:	Mean = 95.9%, s = 0.43% (5 sub samples in duplicate, March 2021)
GC-FID:	Instrument:	Varian CP-3800
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	180 °C (1 min), 30 °C/min to 250 °C (10 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 98.4%, s = 0.06% (10 sub samples in duplicate, February 2014)
Thermogravimetric analysis:		Volatile content < 0.1% and non volatile residue < 0.2% mass fraction (February 2014)
Karl Fischer analysis:		Moisture content ≤ 0.2% mass fraction (February 2014, 2015, 2016, March 2017, February 2018 and January 2021)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	VF-1MS, 30 m × 0.25 mm I.D. × 0.25 µm
	Program:	180 °C (1 min), 30 °C/min to 250 °C (10 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Split ratio:	20/1
	The retention times 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 (12.1 min):	312 (M^+, 26), 270 (12), 252 (100), 237 (15), 214 (15), 213 (15), 210 (13), 209 (12), 198 (10), 167 (11), 165 (13), 155 (10), 141 (16), 115 (10) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/ethyl acetate (7/3) Single spot observed, R_f = 0.40. Visualisation with vanillin
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	2976, 2951, 2878, 2833, 1738, 1655, 1586, 1573, 1438, 1369, 1246, 1023, 890, 766, 694, 556 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX 600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 0.94 (3H, s), 1.32 (1H, dq, *J* = 4.3, 12.4 Hz) 1.44-1.62 (3H, m), 1.73 (1H, m), 1.89 (1H, m), 2.08 (3H, s), 2.27 (1H, m), 2.41 (1H, bt, *J* = 11.8 Hz), 2.46 (2H, t, *J* = 7.5 Hz), 2.51-2.61 (2H, m), 2.75-2.86 (2H, m), 4.80 (1H, dd, *J* = 7.6, 9.4 Hz), 5.77 (1H, s), 6.36 (1H, d, *J* = 9.9 Hz), 6.43 (1H, d, *J* = 9.9 Hz) ppm
	Ethyl acetate estimated at 0.2% mass fraction was observed in the ^{1}H NMR Hexane estimated at 0.2% mass fraction was observed in the ^{1}H NMR	
^{13}C NMR:	Instrument:	Bruker DMX 600
	Field strength:	151 MHz
	Solvent:	$CDCl_3$ (77.2 ppm)
	Spectral data:	δ 14.6, 21.3, 23.3, 24.5, 27.1, 27.7, 31.6, 36.8, 37.6, 45.1, 47.8, 78.6, 123.8, 124.0, 127.5, 141.5, 141.9, 156.3, 171.3, 199.3 ppm
Melting point:		94-96 °C
Microanalysis:	Found:	C = 77.0%; H = 7.8% (February, 2014)
	Calculated:	C = 76.9%; H = 7.7% (Calculated for $C_{20}H_{24}O_3$)