Australian Government
Department of Industry, Innovation and Science

National Measurement Institute

NATA
ACCREDITED FOR TECHNICAL COMPETENCE

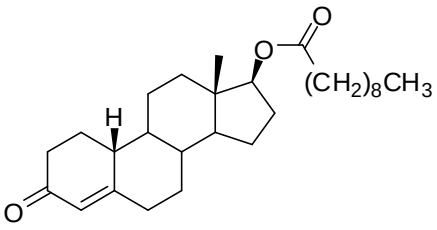
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D684: Nandrolone deconoate

Report ID: D684.2019.01

Chemical Formula: $C_{28}H_{44}O_3$

Molecular Weight: 428.65 g/mol



Property value

Batch No.	CAS No.	Purity (mass fraction)
01-S-04	360-70-3	97.7 ± 2.8%

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

IUPAC name: (17β)-3-Oxoestr-4-en-17-yl decanoate.

Expiration of certification: The property values are valid till 21 May 2024, 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: Creamy white waxy 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 should be used 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: 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 ELS 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.
4 June 2019

This report supersedes any issued prior to 4 June 2019

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 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 elemental microanalysis.

HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus
	Column:	Alltima C-18, 5 µm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	Methanol/MilliQ water (90:10 v/v)
	Flow rate:	1.0 mL/min
	Detector:	Waters 2998 PDA operating at 240 nm Waters ELSD 2424
	Relative peak area of the main component:	
	Re-analysis:	Mean = 97.0%, s = 0.1% (5 sub samples in duplicate, UV at 240 nm June 2014)
	Re-analysis:	Mean = 98.9%, s = 0.1% (5 sub samples in duplicate, ELSD June 2014)
	Re-analysis:	Mean = 98.7%, s = 0.1% (10 sub samples in duplicate, ELSD May 2019)
HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus
	Column:	Alltima C-18, 5 µm (4.6 mm x 150 mm)
	Mobile Phase:	Methanol/MilliQ water (96:4 v/v)
	Flow rate:	0.8 mL/min
	Detector:	Waters 2998 PDA operating at 242 nm Waters ELSD 2424
	Relative peak area of the main component:	
	Initial analysis:	Mean = 97.4%, s = 0.1% (3 sub samples in duplicate, UV at 242 nm June 2001)
	Initial analysis:	Mean = 99.5%, s = 0.1% (3 sub samples in duplicate, ELSD June 2001)
	Re-analysis:	Mean = 97.7%, s = 0.1% (5 sub samples in duplicate, UV at 242 nm, August 2009)
	Re-analysis:	Mean = 98.9%, s = 0.2% (5 sub samples in duplicate, ELSD, August 2009)
GC-FID:	Instrument:	HP5890
	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	100 °C (1 min), 40 °C/min to 300 °C (15 min)
	Injector:	Programmable Cool-On Column
	Carrier:	Helium
	Detector Temp:	325 °C
	Relative peak area of the main component:	
	Initial analysis:	Mean = 97.3%, s = 0.2% (7 sub samples in duplicate, June 2001)
	Re-analysis:	Mean = 98.4%, s = 0.2% (5 sub samples in duplicate, August 2006)
Karl Fischer analysis:		Moisture content < 0.2% mass fraction (July 2006, August 2009 & 2014, May 2019)
Thermogravimetric analysis:		Volatile content not measured due to the volatility of the material. Non volatile residue < 0.2% mass fraction

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP 6890/5973
	Column:	ZB-5, 30 m × 0.25 mm I.D. × 0.25 μm
	Program:	150 °C (1.0 min), 15 °C /min to 300 °C (3 min)
	Injector:	250 °C
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.3 mL/min
	Split ratio:	35/1
	The retention time is reported with the major peaks observed in the mass spectrum. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	11.9 min:	428 (M^+, 52), 274 (100), 257 (57), 256 (65), 155 (43), 110 (56) *m/z*
IR:	Instrument:	Perkin-Elmer FT-IR
	Range:	4000-400 cm^{-1}, KBr disc
	Peaks:	1735, 1677, 1467, 1258, 1214, 1179 cm^{-1}
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/ethyl acetate (4:1) Single spot observed, R_f = 0.44 (3 sub samples)
^{1}H NMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Key spectral data:	δ 0.84 (3H, s), 0.87 (3H, t), 4.62 (1H, dd), 5.82 (1H, s) ppm.
	Isopropanol (0.9%) estimated mass fraction was observed in the ^{1}H NMR.	
^{13}C NMR:	Instrument:	Bruker DMX-600
	Field strength:	151 MHz
	Solvent:	$CDCl_3$ (77 ppm)
	Spectral data:	δ 12.1, 14.1, 22.6, 23.3, 25.1, 26.0, 26.6, 27.4, 29.1, 29.2 (2), 29.4, 30.6, 31.8, 34.6, 35.4, 36.5, 36.6, 40.2, 42.5, 42.7, 49.3, 49.5, 82.2, 124.6, 166.4, 173.9, 199.9 ppm.
Microanalysis:	Found:	C = 78.2%, H = 10.4%. (June 2001)
	Calculated:	C = 78.5%, H = 10.3% (Calculated for $C_{28}H_{44}O_3$)
Melting point:		24-31 ºC by DSC