



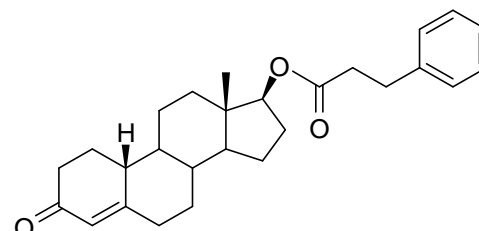
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA S029: Nandrolone phenylpropionate

Report ID: S029.2020.04 (Bottled 150907)

Chemical Formula: C₂₇H₃₄O₃

Molecular Weight: 406.6 g/mol



Property value

Batch No.	CAS No.	Purity estimate
14-S-06	62-90-8	99.2 ± 1.3%

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

Expiration of certification: The property values are valid till 11 June 2025, 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: 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 °C 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%.

Stability: In the absence of long term stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated from stability trials conducted on similar materials by NMI Australia over the last ten years. The measurement uncertainty at the 95% confidence interval also includes a stability component determined from accelerated stability trials conducted at 40 °C and 75% humidity for a 14 day period.

The long-term stability of the compound in solution has not been examined.

Stability: This material has demonstrated stability over a minimum period of three years. The measurement uncertainty includes a component for long term stability at the recommended storage conditions.

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 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 HPLC with UV detection, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR spectroscopy. The purity value is calculated as per Equation 1.

$$\text{Purity} = (100\% - I_{\text{ORG}}) \times (100\% - I_{\text{VOL}} - I_{\text{NVR}}) \quad \text{Equation 1}$$

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

Supporting evidence is provided by qualitative headspace GC-MS analysis of occluded solvents and elemental microanalysis.

HPLC: Instrument: Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
 Column: Alltima C18, 5 μm (4.6 mm x 150 mm)
 Column oven: 40 $^{\circ}\text{C}$
 Mobile Phase: Methanol/Milli Q water (85:15)
 Flow rate: 1.0 mL/min
 Detector: Shimadzu SPD-M20A PDA operating at 236 nm
 Relative peak area of the main component:
 Initial analysis: Mean = 99.4%, s = 0.02% (10 sub samples in duplicate, July 2014)

HPLC: Instrument: Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
 Column: Alltima C18, 5 μm (4.6 mm x 150 mm)
 Column oven: 40 $^{\circ}\text{C}$
 Mobile Phase: Methanol/Milli Q water (79:21)
 Flow rate: 1.0 mL/min
 Detector: Shimadzu SPD-M20A PDA operating at 236 nm
 Relative peak area of the main component:
 Initial analysis: Mean = 99.3%, s = 0.02% (5 sub samples in duplicate, June 2020)

HPLC: Instrument: Waters Model 1525 Binary pump, 717 plus auto sampler
 Column: Alltima C18, 5 μm (4.6 mm x 150 mm)
 Column oven: 40 $^{\circ}\text{C}$
 Mobile Phase: Methanol/Milli Q water (79:21)
 Flow rate: 1.0 mL/min
 Detector: Waters 2998 PDA operating at 241 nm
 Relative peak area of the main component:
 Initial analysis: Mean = 99.3%, s = 0.02% (5 sub samples in duplicate, July 2015)
 Re-analysis: Mean = 99.3%, s = 0.01% (5 sub samples in duplicate, June 2016)
 Re-analysis: Mean = 99.4%, s = 0.01% (5 sub samples in duplicate, June 2017)

Karl Fischer analysis: Moisture content \leq 0.1% mass fraction (July 2014 and 2015)
 Moisture content 0.13% mass fraction (June 2016)
 Moisture content < 0.1% mass fraction (June 2017)
 Moisture content 0.1% mass fraction (June 2020)

Thermogravimetric analysis: Volatile content < 0.1% and non volatile residue < 0.2% mass fraction (August 2014)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	HP6890/5973
	Column:	TG1-MS, 30 m x 0.25 mm I.D. x 0.25 μ m
	Program:	250 °C (1 min), 30 °C/min to 300 °C (15 min)
	Injector:	250 °C,
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 <i>m/z</i>
	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 (12.0 min):	406 (M^+ , 1), 257 (100), 239 (12), 147 (20), 135 (11), 133 (11), 105 (40), 91 (44) <i>m/z</i>
HS-GC-MS:	Instrument:	Agilent 6890/5973/G1888
	Column:	DB-624, 30 m x 0.25 mm I.D. x 1.4 μ m
	Program:	50 °C (5 min), 7 °C/min to 120 °C, 15 °C/min to 220 °C (8.3 min)
	Injector:	150 °C
	Split ratio:	50/1
	Transfer line temp:	280 °C
	Carrier:	Helium, 1.2 mL/min
	Solvents detected:	Acetone
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Hexane/ethyl acetate (1:1) Single spot observed, $R_f = 0.6$. Visualisation with UV at 254 nm.
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1} , neat
	Peaks:	2942, 2918, 2873, 2855, 1727, 1670, 1259, 1144, 1045, 876, 746, 702 cm^{-1}
¹ H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	CDCl ₃ (7.26 ppm)
	Spectral data:	δ 0.79 (3H, s), 0.85 (1H, m), 1.02-1.11 (2H, m), 1.14-1.26 (2H, m), 1.29-1.39 (2H, m), 1.43-1.59 (2H, m), 1.62-1.70 (2H, m), 1.80-1.84 (2H, m), 2.07 (1H, m), 2.15 (1H, m), 2.21-2.29 (3H, m), 3.38-2.43 (1H, m), 2.49 (1H, m), 2.63 (2H, t, $J = 7.5$ Hz), 2.95 (2H, t, $J = 7.8$ Hz), 4.61 (1H, dd, $J = 8.0, 9.1$ Hz), 5.82 (1H, s), 7.18-7.21 (3H, m), 7.26-7.29 (2H, m) ppm
¹³ C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	CDCl ₃ (77.2 ppm)
	Spectral data:	δ 12.2, 23.5, 26.1, 26.7, 27.6, 30.7, 31.2, 35.6, 36.2, 36.7, 40.3, 42.7, 42.8, 49.5, 49.6, 82.7, 124.8, 126.4, 128.4, 128.6, 140.6, 166.6, 173.1, 200.1 ppm
Melting point:		97-98 °C
Microanalysis:	Found:	C = 79.8%; H = 8.4% (August, 2014)
	Calculated:	C = 79.8%; H = 8.4% (Calculated for C ₂₇ H ₃₄ O ₃)