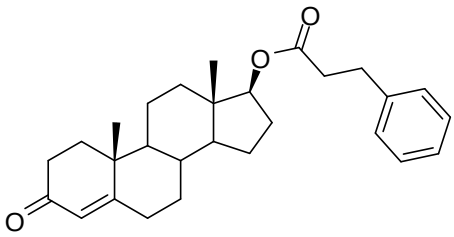
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

Report ID: S030.2018.01 (Bottled 160511)
This batch of bottles was prepared from the bulk material on 11th May 2016.

Compound Name: **Testosterone phenylpropionate**
Collection Number: S030
Chemical Formula: $C_{28}H_{36}O_3$
CAS Number: 1255-49-8
Structure:

Description: Off white solid
Batch Number: 14-S-07
Molecular Weight: 420.6
Release date: 28th April 2015



Synonyms: 17β-(1-Oxo-3-phenylpropoxy)-androst-4-en-3-one
Testosterone, hydrocinnamate
3-Oxoandrost-4-en-17β-yl β-phenylpropionate
Testosterone 17-phenylpropionate

Purity (mass fraction): 98.1 ± 1.4% (95% coverage interval)

The purity value was determined by quantitative NMR analysis using a combination of the one proton singlet at 5.62 ppm and the one proton triplet at 4.49 ppm against a certified internal standard of dimethyl terephthalate. Supporting evidence is provided by HPLC with UV/Vis detection at 210 nm, thermogravimetric analysis, Karl Fischer analysis, ^{1}H NMR, and elemental microanalysis.

HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	Alltima C-18, 5 μm (4.6 mm x 150 mm)
	Column oven:	35 °C
	Mobile Phase:	Acetonitrile/MilliQ water (85:15)
	Flow rate:	1 mL/min
	Detector:	Shimadzu SPD-M20A PDA operating at 210 nm
	Relative peak area response of main component:	
	Initial analysis:	Mean = 99.4%, s = 0.02% (10 sub samples in duplicate, March 2015)
	Re-analysis:	Mean = 99.4%, s = 0.04% (5 sub samples in duplicate, April 2016)
	Re-analysis:	Mean = 99.5%, s = 0.01% (5 sub samples in duplicate, May 2017)
	Re-analysis:	Mean = 99.4%, s = 0.06% (5 sub samples in duplicate, May 2018)
Thermogravimetric analysis:		Volatile content 0.1% and non volatile residue 0.3% mass fraction (March 2015)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (March 2015 and April 2016) Moisture content 0.15% mass fraction (May 2017) Moisture content 0.39% mass fraction (April 2018)
QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz Solvent: d_6-DMSO (2.50 ppm)
	Internal standard:	Dimethyl terephthalate (100% mass fraction)
	Initial analysis:	Mean (5.62 ppm) = 98.2%, s = 0.3% (5 sub samples, April 2015)
	Initial analysis:	Mean (4.49 ppm) = 98.1%, s = 0.3% (5 sub samples, April 2015)

Spectroscopic and other characterisation data

LC-MS:	Instrument:	Waters 2695 (HPLC)/Micromass Quatro	
	Column:	Alltima, 100 mm × 4.6 mm I.D. × 5.0 μm	
	Column temp:	40 °C	
	Solvent system:	0.2 percent formic acid buffered to pH 3 [0.2% v/v], acetonitrile [85% v/v], MilliQ water [14.8% v/v]	
	Flow rate:	1 mL/min	
	Sample prep:	1000 μg/g in acetonitrile/MilliQ water (85:15)	
	Injection volume:	10 μL	
	Ionisation mode:	Electrospray positive ion	
	Capillary voltage:	3.5 kV	Cone voltage: 10 V
	Source temp:	130 °C	Desolvation gas temperature: 350 °C
	Cone gas flow rate:	26 L/hr	Desolvation gas flow rate: 751 L/hr
	The retention time of testosterone phenylpropionate is reported along with the major peak in the mass spectrum. The latter is reported as a mass/charge ratio.		
	6.21 min:	421.3 ($M+H^+$) m/z	
TLC:	Conditions:	Kieselgel 60F_{254}. Hexane/acetone (4/1) Single spot observed, R_f = 0.40. Visualisation with UV at 254 nm	
IR:	Instrument:	Bruker Alpha FT-IR	
	Range:	4000-400 cm^{-1}, neat	
	Peaks:	2942, 1733, 1671, 1416, 1290, 1229, 1162, 1006, 863, 752, 701, 518 cm^{-1}	
^{1}H NMR:	Instrument:	Bruker Avance-III	
	Field strength:	500 MHz	Solvent: CD_3OD (3.31 ppm)
	Spectral data:	δ 0.80 (3H, s), 0.94-1.17 (4H, m), 1.23 (3H, s), 1.32-1.49 (3H, m), 1.56-1.72 (5H, m), 1.88 (1H, m), 2.05-2.14 (2H, m), 2.26-2.32 (2H, m), 2.44-2.51 (2H, m), 2.64 (2H, t, *J* = 7.5 Hz), 2.92 (2H, t, *J* = 7.5 Hz), 4.57(1H, dd, *J* = 8.0, 9.5 Hz), 5.71 (1H, s), 7.16-7.27 (5H, m) ppm	
	Hexane estimated at 0.2% mass fraction was observed in the ^{1}H NMR Ethyl acetate estimated at 0.04% mass fraction was observed in the ^{1}H NMR		
^{13}C NMR:	Instrument:	Bruker Avance-III	
	Field strength:	125 MHz	Solvent: CD_3OD (49.0 ppm)
	Spectral data:	δ 12.4, 17.7, 21.6, 24.4, 28.4, 32.1, 32.8, 33.8, 34.7, 36.6, 36.8, 36.9, 37.8, 40.0, 43.7, 51.5, 55.2, 84.0, 124.2, 127.3, 129.4, 129.5, 141.8, 174.7, 174.9, 202.3 ppm	
Melting point:		115-116 °C	
Microanalysis:		Found: C = 79.9%; H = 8.8% (March 2015) Calc: C = 80.0%; H = 8.6% (Calculated for $C_{28}H_{36}O_3$)	

Expiration of certification
The property values are valid till 31st May 2021, 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.

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

This material has been given a shelf life of three years from the date of re-certification. The material will be re-tested on an annual basis to ensure that the property values are still valid. In the event a product fails the stability trial, notification will be sent to all impacted customers.

In the absence of 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 10 years.

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.

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.

Intended use
For ***in vitro*** laboratory analysis only.

Caution
Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice
Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 7 June, 2018.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 7th June, 2018.