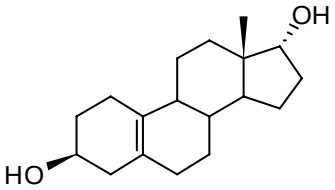
Australian Government

National Measurement Institute

REFERENCE MATERIAL ANALYSIS REPORT

Report ID: D926.2009.02

Compound Name: **5(10)-Estrene-3β,17α-diol**
Collection No: D926
Chemical Formula: $C_{18}H_{28}O_2$
CAS No: 268734-48-1
Structure:



Description: White solid
Batch No: 08-S-02
Molecular Weight: 276.4
Batch production completed: January 2008

Synonyms: (3β,17α)-Estr-5(10)-ene-3,17-diol

Purity (mass fraction): 97.0 ± 1.0 % (95 % confidence interval)

Purity estimate obtained by subtraction from 100% of total impurities by GC-FID, TGA, Karl Fischer and ^{1}H NMR.

GC-FID:	Instrument:	Varian CP-3800	
	Column:	VF-1, 30 m × 0.32 mm I.D. × 0.25 µm	
	Program:	180 °C (1 min), 40 °C/min to 250 °C (10 min), 40 °C/min to 300 °C (2 min)	
	Injector:	200 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative peak area response of main component:		
	Initial analysis:	Mean = 99.6%, s = 0.001 (7 sub samples in duplicate, February 2008)	
GC-FID:	Instrument:	Agilent 6890N	
	Column:	HP-1, 29.5 m × 0.32 mm I.D. × 0.25 µm	
	Program:	190 °C (21 min), 30 °C/min to 300 °C (1 min)	
	Injector:	250 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative peak area response of main component:		
	Initial analysis:	Mean = 99.3%, s = 0.005 (5 sub samples in duplicate, February 2009)	

GC-MS:	Parent compound:		
	Instrument:	Agilent 6890 / 5973	
	Column:	Ultra 2, 17 m x 0.20 mm I.D. x 0.25 µm	
	Program:	80 °C (0.5 min), 50 °C/min to 200 °C (0.5 min), 15 °C/min to 310 °C (5 min)	
	Injector:	200 °C	Transfer line temp: 300 °C
	Carrier:	Helium, 1.0 mL/min	Split ratio: 10/1
	Bis-TMS derivative:		
	Instrument:	Agilent 6890 / 5973	
	Column:	Ultra 1, 17m x 0.2mm I.D.x 0.11µm	
	Program:	187 °C (0.2 min), 3 °C/min to 238 °C, 10 °C/min to 265 °C, 30 °C/min to 310 °C (2 min)	
	Injector:	250 °C	Transfer line temp: 300 °C
	Carrier:	Helium, 1.0 mL/min	Split ratio: 12/1
	The retention times of the parent compound and *bis*-TMS derivative are 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 (6.4 min):	276 (M^+, 48), 258 (61), 240 (69), 225 (100), 199 (48), 185 (17), 159 (21), 145 (32), 131 (37), 117 (28), 105 (37), 91 (61), 79 (28) m/z.	
	Bis-TMS (7.0 min):	420 (M+, 4), 405 (4), 330 (100), 240 (55), 225 (43), 212 (11), 199 (30), 145 (21), 129 (32), 91 (24), 73 (68) m/z	
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform / ethyl acetate (2/1). Single spot observed, R_f = 0.48 Visualisation with vanillin dip	
IR:	Instrument:	Biorad FTS300MX FT-IR.	
	Range:	4000-400cm^{-1}, KBr pellet.	
	Peaks:	3352, 2916, 1447, 1375, 1296, 1096, 1034, 970 cm^{-1}	
^{1}H NMR:	Instrument:	Bruker DMX600	
	Field strength:	600 MHz	Solvent: CD_3OD
	Spectral data:	δ 0.68 (3H, s), 1.16-1.33 (4H, m), 1.42-1.54 (3H, m), 1.56-1.77 (6H, m), 1.79-1.86 (2H, m), 1.88-1.96 (2H, m), 1.99-2.19 (3H, m), 2.29 (1H, bm), 3.65 (1H, d, *J* =6.1 Hz), 3.94 (1H, m) ppm.	
^{13}C NMR:	Instrument:	Bruker DMX600	
	Field strength:	151 MHz	Solvent: CD_3OD
	Spectral data:	δ 17.8, 24.1, 25.1, 26.3, 29.0, 31.2, 32.4, 32.8, 33.2, 39.9, 40.8, 46.9, 47.4, 48.7, 66.7, 80.7, 125.6, 130.8 ppm.	
Melting point:		148-154 °C	
Microanalysis:		Found: C = 76.9 %, H = 10.8 % (February 2008) Calc: C = 78.2 %, H = 10.2 % (for $C_{18}H_{28}O_2$)	
Thermogravimetric analysis:		Volatile content c.a. 2% and non volatile residue c.a. 0.2 % mass fraction (February 2008 & 2009).	
Karl Fischer analysis:		Moisture content c.a. 2% mass fraction (February 2008 & 2009).	

Expiration of certification
The property values are valid till 25th February 2012, 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/ampoules 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 3 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% confidence 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 GC-FID on 5 randomly selected 1-2 mg samples of the material. The material was judged to be 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: 2 August, 2012.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 18th July 2012.

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