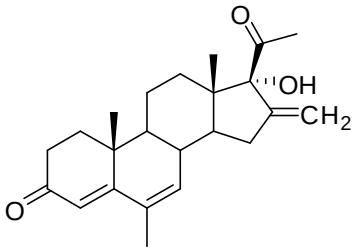
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA D655: Melengesterol



Report ID: D655.2017.03

Chemical Formula: $C_{23}H_{30}O_3$

Molecular Weight: 354.5 g/mol

Property value

Batch No.	CAS No.	Purity estimate
00-AV-04	5633-18-1	99.5%

IUPAC name: 17-Hydroxy-6-methyl-16-methylenepregna-4,6-diene-3,20-dione**.**

Expiration of certification: The property values are valid till 27 March 2022, 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: Pale yellow crystals prepared by synthesis, 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: This material has demonstrated stability over a minimum period of five 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 HPLC with ELS 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.
25 June 2021

This report supersedes any issued prior to 25 June 2021

NATA logo notice: Accredited for compliance with ISO 17034. Accreditation No. 198 / Corporate Site No. 14214. 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 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 qualitative elemental microanalysis.

HPLC:	Column:	Alltima C-18, 5 μm (4.6 mm x 150 mm)
	Mobile Phase:	Methanol/water (75:25)
	Flow rate:	1.0 mL/min
	Detector:	ELSD
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 99.8%, s = 0.33% (10 sub samples in duplicate, August 2000)
	Re-analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, April 2007)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, April 2012)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 sub samples in duplicate, March 2017)
Karl Fischer analysis:		Moisture content ≤ 0.2% mass fraction (April 2012 and March 2017)
Thermogravimetric analysis:		Volatiles content 0.3% mass fraction (September 2000), <0.1% mass fraction (April 2007) and non-volatile residue < 0.2% mass fraction (September 2000 & April 2007)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP6890/5973
	Column:	HP Ultra 1, 17 m x 0.20 mm I.D. x 0.11 μm
	Program:	170 °C (0.5 min), 10 °C/min to 300 °C (3 min)
	Injector:	260 °C
	Split ratio:	40/1
	Transfer line temp:	300 °C
	Carrier:	Helium
	The retention time of *tris*-TMS derivative is reported with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage peak intensity relative to the intensity of the base peak.	
	Tris-TMS (10.0 min):	570 (M^+, 28), 465 (8), 313 (13), 243 (11), 147 (12), 73 (100) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/Ethyl acetate (4:1) Single spot observed, R_f = 0.33 (5 samples)
IR:	Instrument:	Perkin-Elmer FT-IR
	Range:	4000-400 cm^{-1}, KBr disc
	Peaks:	3491, 1706, 1651, 1621, 1575, 1544, 1278, 1232, 898 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Spectral data:	δ 0.86 (3H, s), 1.09 (3H, s), 1.83 (3H, s), 2.31 (3H, s), 5.10 (1H, s), 5.31 (1H, s), 5.86 (1H, s), 5.92 (1H, s) ppm
^{13}C NMR:	Instrument:	Bruker DMX-600
	Field strength:	151 MHz
	Solvent:	$CDCl_3$ (77.5 ppm)
	Spectral data:	δ 15.5, 16.3, 19.8, 19.9, 28.7, 29.9, 32.4, 33.5, 33.8, 36.1, 36.2, 46.5, 49.3, 50.1, 90.0, 114.3, 121.2, 131.3, 137.9, 152.2, 164.1, 199.9, 210.8 ppm
Melting point:		205-208 °C (decomp)
Microanalysis:	Found:	C = 77.9%; H = 8.5% (August 2000)
	Calculated:	C = 77.9%; H = 8.5% (Calculated for $C_{23}H_{30}O_3$)