Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

NATA ACCREDITED FOR TECHNICAL COMPETENCE

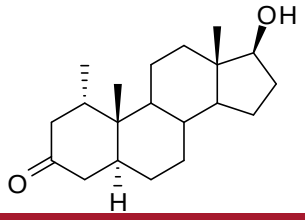
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA S037: Mesterolone

Report ID: S037.2020.01 (Bottled 160623)

Chemical Formula: $C_{20}H_{32}O_2$

Molecular Weight: 304.5 g/mol



Property value

Batch No.	CAS No.	Purity estimate
15-S-12	1424-00-6	99.5 %

IUPAC name: (1α,5α,17β)-17-Hydroxy-1-methylandrostan-3-one.

Expiration of certification: The property values are valid till 24 July 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. 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.

Description: White crystalline 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: 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 10 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 GC-FID on nine 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.
1 September 2020

This report supersedes any issued prior to 1 September 2020.

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 purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, 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 headspace GC-MS analysis of occluded solvents and elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800 or Agilent 7890
	Column:	HP-5, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	180 °C (1 min), 30 °C/min to 250 °C (10 min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.7%, s = 0.02% (9 sub samples in duplicate, October 2015)
	Re-analysis:	Mean = 99.6%, s = 0.05% (5 sub samples in duplicate, November 2016)
	Re-analysis:	Mean = 99.6%, s = 0.01% (5 sub samples in duplicate, September 2017)
	Re-analysis:	Mean = 99.5%, s = 0.01% (5 sub samples in duplicate, July 2020)
GC-FID:	Instrument:	Varian CP-3800
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	180 °C (1 min), 30 °C/min to 250 °C (10 min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.7%, s = 0.03% (9 sub samples in duplicate, October 2015)
Karl Fischer analysis:		Moisture content < 0.1% mass fraction (October 2015, August 2016, September 2017 and July 2020)
Thermogravimetric analysis:		Volatile content < 0.2% and non-volatile residue < 0.2% mass fraction (October 2015)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890/5973
	Column:	HP-1MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	150 °C (1 min), 10 °C/min to 270 °C (5 min), 30 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Split ratio	20/1
	Transfer line temp:	300 °C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 *m/z*
	Bis-TMS derivative:	
	Instrument:	Agilent 6890/5973
	Column:	HP-1MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	200 °C (1 min), 2 °C/min to 270 °C (1 min), 30 °C/min to 300 °C (2 min)
	Injector:	250 °C
	Split ratio	20/1
	Transfer line temp:	300 °C
	Carrier:	Helium
	Scan range:	50-550 *m/z*
	The retention times of the parent compound and *bis*-TMS derivatives are reported along 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 (13.9 min):	304 (M^+, 84), 245 (19), 218 (100), 201 (74), 185 (22), 174 (26), 159 (41), 123 (27), 105 (41), 81 (42), 67 (40) *m/z*
	Bis-TMS (21.3 min):	448 (M^+, 6), 433 (17), 157 (47), 141 (100), 73 (34) *m/z*
	Bis-TMS (21.5 min):	448 (M^+, 10), 433 (11), 157 (50), 141 (100), 73 (44) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/acetone (4/1) Single spot observed, R_f = 0.63. Visualisation with vanillin stain
IR:	Instrument:	Bruker Alpha FT-IR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	3414, 3364, 2913, 1705, 1419, 1380, 1066, 528cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	MeOH-d_4 (3.31ppm)
	Key spectral data:	δ 0.76 (3H, s), 0.87 (3H, d, *J* = 7.2 Hz), 0.89 (1H, m), 0.97-1.09 (3H, m), 1.18 (3H, s), 1.28 (1H, m), 1.33-1.64 (7H, m), 1.72 (1H, m), 1.75-1.87 (2H, m), 1.94-2.02 (3H, m), 2.17 (1H, m), 2.36 (1H, m), 2.83 (1H, dd, *J* = 6.2, 14.4 Hz), 3.58 (1H, t, *J* = 8.7 Hz) ppm
^{13}C NMR	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	MeOH-d_4 (49.0 ppm)
	Spectral data:	δ 11.7, 14.7, 15.1, 21.3, 24.3, 29.9, 30.6, 32.3, 36.9, 37.9, 39.1, 40.9, 41.5, 44.2, 45.8, 46.7, 50.1, 52.2, 82.4, 215.4 ppm
Melting point:		209 – 210ºC
Microanalysis:	Found:	C = 79.0%; H = 10.7% (October 2015)
	Calculated:	C = 78.9%; H = 10.6% (Calculated for $C_{20}H_{32}O_2$)