Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

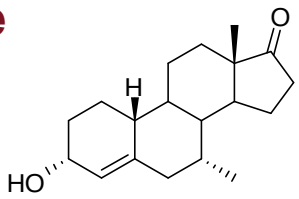
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA S050: 7α-Methyl-estr-4-ene-3α-ol-17-one

Report ID: S050.2022.01 (Ampouled 190815)

Chemical Formula: $C_{19}H_{28}O_2$

Molecular Weight: 288.42 g/mol



Property value

Batch No.	CAS No.	Mass per ampoule
19-S-05	**Not available**	**966 μg ± 15 μg**

IUPAC name: 7α-Methyl-estr-4-ene-3α-ol-17-one.

Expiration of certification: The property values are valid till 10 June 2025, i.e. three years from the date of 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 ampoules that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials.

Description: The compound is supplied as a dried aliquot in a sealed ampoule and is intended for a single use to prepare a standard solution containing S050. Material was prepared by synthesis, and certified for identity and purity by NMIA.

Intended use: This reference material should be used for qualitative analysis only.

Instructions for use: Open the ampoule and carefully rinse the interior at least three times with a suitable organic solvent (e.g. acetonitrile). This will transfer 966 μg of anhydrous 7α-methyl-estr-4-ene-3α-ol-17-one.

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: 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 seven randomly selected ampoules 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 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.
24 October 2022

This report supersedes any issued prior to 24 October 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:

GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-1 or HP-5, 30 m × 0.32 mm I.D. × 0.25 µm
	Program:	180 °C (1 min), 20 °C/min to 240 °C (10 min), 20 °C/min to 280 °C (8 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component as the *mono*-TMS derivative:	
	Initial analysis:	Mean = 96.6%, s = 0.01% (7 ampoules in duplicate, October 2019)
	Re-analysis:	Mean = 97.2%, s = 0.02% (5 ampoules in duplicate, September 2020)
	Re-analysis:	Mean = 96.9%, s = 0.01% (5 ampoules in duplicate, August 2021)
	Re-analysis:	Mean = 96.9%, s = 0.02% (5 ampoules in duplicate, June 2022)

The following analytical data was obtained on the bulk material subsequently used in the preparation of the ampoules.

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 elemental microanalysis.

GC-FID:	Instrument:	Varian CP-3800
	Column:	HP-1, 29.4 m × 0.32 mm I.D. × 0.25 µm
	Program:	180 °C (1 min), 20 °C/min to 240 °C (10 min), 20 °C/min to 300 °C (8 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component as the *mono*-TMS derivative:	
	Initial analysis:	Mean = 96.8%, s = 0.2% (10 sub samples in duplicate, May 2019)
Karl Fischer analysis:		Moisture content 0.2% mass fraction (June 2019)
Thermogravimetric analysis:		Volatiles content 0.4% and non-volatile residue 0.3% mass fraction (May 2019)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP6890/5973
	Column:	DB-5MS 30 x 0.25 mm I.D. x 0.25 μm
	Program:	180 °C (1 min), 20 °C/min to 240 °C (10 min), 20 °C/min to 280 °C (8 min)
	Injector:	200 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium 1.0 mL/min
	Scan range:	50-550 *m/z*
	TMS derivative:	
	Instrument:	HP 6890/5973
	Column:	DB-5MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	180 °C (1 min), 20 °C/min to 240 °C (10 min), 20 °C/min to 280 °C (8 min)
	Injector:	200 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium 1.0 mL/min
	Scan range:	50-550 *m/z*
	The retention times of the parent compound and the mono-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 (10.87 min):	288 (M^+, 26), 270 (82), 255 (16), 159 (18), 149 (24), 145 (27), 143 (22), 131 (22), 91 (100), 81 (30), 79 (46), 41 (41) *m/z*
	Mono-TMS (11.3 min):	360 (M^+, 15), 270 (32), 149 (10), 129 (11), 105 (16), 91 (37), 75(100), 73 (17) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Dichloromethane/Diethyl ether (4:1) Single spot observed, R_f = 0.5
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1}, neat
	Peaks:	3506, 2947, 2910, 2883, 2850, 1722, 1454, 1405, 1344, 1251, 1177, 1089, 1015, 979, 952, 847, 676, 584 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, d, *J* = 7.0 Hz), 0.91 (3H, s), 1.04 (1H, m), 1.15-1.27 (2H, m), 1.36-1.43 (3H, m), 1.50-1.59 (3H, m), 1.67 (1H, m), 1.75-1.80 (2H, m), 1.85 (1H, m), 1.90-1.99 (3H, m), 2.05-2.13 (2H, m), 2.30 (1H, dd, *J* = 3.5, 13.5 Hz), 2.45 (1H, dd, *J* = 8.5, 19.0 Hz), 4.12 (1H, m), 5.58 (1H, m) ppm
		Ethyl acetate and 7α-methylestr-4-ene-3β-ol-17-one were estimated at < 0.2% and 3% mass fraction respectively by ^{1}H NMR.
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	$CDCl_3$ (77.16 ppm)
	Spectral data:	δ 12.7, 13.9, 21.3, 22.8, 26.4, 29.9, 30.5, 31.5, 35.8, 42.6, 42.6, 42.8, 42.9, 47.3, 48.1, 64.6, 124.4, 142.4, 221.2 ppm
Melting point:		153-156°C
Microanalysis:	Found:	C = 78.9%; H = 9.6% (June, 2019)
	Calculated:	C = 79.1%; H = 9.8% (Calculated for $C_{19}H_{28}O_2$)