

# Australian Government



# **National Measurement Institute**

# **REFERENCE MATERIAL PRODUCT INFORMATION SHEET**

## Report ID: D832.2018.01 (Ampouled 151013)

This batch of ampoules was prepared from the bulk material on 13<sup>th</sup> October 2015.

Compound Name: **5\alpha-Androst-1-ene-3\alpha-ol-17-one** Collection Number: D832 Chemical Formula: C<sub>19</sub>H<sub>28</sub>O<sub>2</sub> CAS Registry Number: 38859-37-9 Structure: Description: White powder Batch Number: 03-S-06 Molecular Weight: 288.4 Release date: March 2004 Metabolite of 1-testosterone & 1-androstendione



Synonyms:  $5\alpha$ -Androst-1-ene- $3\alpha$ -ol-17-one  $3\alpha$ -Hydroxy- $5\alpha$ -androst-1-en-17-one

Purity by GC-FID:

83.8%, s = 0.13% (n = 7)

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 D832. Approximately, 200  $\mu$ g of bulk material was dispensed into each ampoule. Open the ampoule and carefully rinse the interior at least three times with a suitable organic solvent (chloroform).

This reference material has NOT been certified for purity as a mass fraction. Accordingly, this material should be considered for use in qualitative analysis only.

GC-FID	Instrument:	Varian CP-3800			
	Column:	HP-5, 30 m $\times$ 0.32 mm I.D. $\times$ 0.25 $\mu$ m			
	Program	140 °C (1 min), 30 °C /min to 230 °C (13 min), 30 °C /min to 300 °C (3 min)			
	Injector:	250 °C	Detector Temp: 320 °C		
	Carrier:	Helium	Split ratio: 20/1		
	Relative peak area response of main component:				
	Initial analysis:	Mean = $83.8\%$ , s = $0.13\%$ (7 ampoules in duplicate, October 2015)			
	Re-analysis:	Mean = 84.0%, s =	0.09% (5 ampoules in duplicate, September 2016)		
	Re-analysis:	Mean = 83.6%, s =	0.07% (5 ampoules in duplicate, August 2017)		
	Re-analysis:	Mean = $84.4\%$ , s =	0.07% (5 ampoules in duplicate, September 2018)		

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

Purity estimate obtained from a combination of traditional analytical techniques. The purity estimate by traditional analytical techniques was obtained by subtraction from 100% of total impurities by GC-FID and thermogravimetric analysis and <sup>1</sup>H NMR.

GC-FID	Data derived from injections of native D832 and <i>bis</i> -trimethylsilyl derivatives:					
	Instrument:	Agilent6890N				
	Column:	HP-1, 30 m × 0.32 mm I.D. × 0.25 $\mu$ m				
	Program	140 °C (1 min), 40 °C /min to 190 °C (20 min), 40 °C /min to 300 °C (3 min)				
	Injector:	250 °C	Detector Temp: 320 °C			
	Carrier:	Helium	Split ratio: 20/1			
	Relative peak area response of main component:					
	Initial analysis:	Mean = $84.8\%$ , s = $1.0\%$ (5 samples in duplicate, March 2004)				
Thermogravimetric analysis:		Volatiles content 1.3% and non-volatile residue 0.3% mass				
		fraction (March 2004 & August 2005)				

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# Spectroscopic and other characterisation data

GC-MS:	<i>Bis</i> -trimethylsilyl derivative:			
	Instrument:	Agilent 6890/5973		
	Column:	Ultra 1, 17 m × 0.2 mm I.D. × 0.11 μm		
	Program:	189 °C (0.2 min) 3 °C /min to 240 °C, 10 °C /min to 265,		
		30 °C/min to 310 °C		
	Injector:	250 °C	Transfer line temp: 300 °C	
	Carrier:	Helium 1.0 mL/min	Split ratio: 14/1	
	The retention time of the <i>bis</i> -TMS derivative is reported along with the major peaks in the mass spectrum. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.			
	9.5 min:	432 (M <sup>+</sup> , 62), 417 (63), 327	(20), 290 (24), 275 (41), 169 (23), 73 (100) <i>m</i> / <i>z</i>	
IR:	Instrument: Range:	FT-IR, Biorad FTS3000MX 4000-400 cm <sup>-1</sup> KBr		
	Peaks:	3530, 3025, 2927, 1734, 1454, 1376, 1260, 1053, 1005, 750 cm <sup>-1</sup>		
<sup>1</sup> H NMR:	Instrument:	Bruker DMX-500		
	Field strength:	500 MHz	Solvent: $CDCl_3$ (7.26 ppm)	
	Key spectral data:	δ 0.83 (3H, s), 0.88 (3H, s),	4.11 (1H, t), 6.66 (1H, m), 6.06 (1H, d) ppm	
<sup>13</sup> C NMR:	Instrument:	Bruker DMX-500		
	Field strength:	126 MHZ	Solvent: CDCl <sub>3</sub> (77.2 ppm)	
	Spectral data:	8 13.8, 13.9, 20.4, 21.8, 27.   51.1, 51.5, 64.3, 126.3, 139	/, 30.8, 31.5, 34.7, 35.3, 35.8, 38.1, 39.0, 47.9, .9, 221.1 ppm	



# **Expiration of certification**

The property values are valid till 11<sup>th</sup> of September 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 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.

## 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.

#### **Recommended storage**

When not in use this material should be stored at or below 4 °C in a closed container in a dry, dark area.

# Intended use

This reference material should be used for qualitative 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: 21 September, 2018.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 21st September, 2018



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