Australian Government
Department of Industry, Innovation and Science

National Measurement Institute

CIPM MRA

NATA
ACCREDITED FOR TECHNICAL COMPETENCE

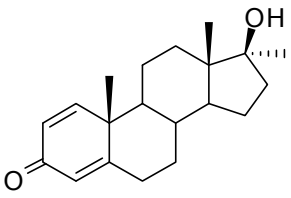
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D630: Methandienone

Report ID: D630.2018.02

Chemical Formula: $C_{20}H_{28}O_2$

Molecular Weight: 300.4 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
00-S-04	72-63-9	98.1 ± 0.3%

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit (k = 2).

IUPAC name: (17β)-17-Hydroxy-17-methylandrosta-1,4-dien-3-one

Expiration of certification: The property values are valid till 30 April 2023, 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:** White crystals prepared by 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 certified reference material is suitable for use as a primary calibrator.

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.

Metrological traceability: The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance approach all impurities are quantified as a mass fraction and subtracted from 100%. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

Stability: This material has demonstrated stability over a minimum period of five years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual 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 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.
11 February 2020

This report supersedes any issued prior to 11 February 2020

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified 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 elemental microanalysis.

GC-FID:	Instrument:	Agilent 6890N	
	Column:	HP-1 Capillary, 30 m × 0.32 mm I.D. × 0.25 µm	
	Program:	180 °C (1 min), 15 °C/min to 300 °C (5 min)	
	Injector:	250 °C	Detector Temp: 320 °C
	Carrier:	Helium	Split ratio: 20/1
	Relative mass fraction of the main component:		
	Initial analysis:	Mean = 98.6%, s = 0.08% (10 sub samples in duplicate, March 2000)	
	Re-analysis:	Mean = 98.4%, s = 0.01% (5 sub samples in duplicate, June 2008)	
	Re-analysis:	Mean = 98.3%, s = 0.01% (5 sub samples in duplicate, May 2013)	
	Re-analysis:	Mean = 98.2%, s = 0.03% (5 sub samples in duplicate, April 2018)	
Karl Fischer analysis:		Moisture content 0.10% mass fraction (August 2006) Moisture content 0.16% mass fraction (May 2008) Moisture content 0.23% mass fraction (May 2013) Moisture content 0.27% mass fraction (May 2018)	
Thermogravimetric analysis:		Volatiles content and non-volatile residue < 0.3% mass fraction (February 2000)	

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	HP6890/5973
	Column:	HP Ultra 2, 17 m × 0.22 mm I.D. × 0.11 µm
	Program:	170 °C, 3 °C/min to 234 °C, 10 °C/min to 265 °C (3 min)
	Injector:	280 °C Split ratio: 15/1
	Transfer line temp:	300 °C
	Carrier:	Helium
	The retention time of the material is reported 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.	
	Parent (8.1 min):	300 (M^+, 7), 282 (10), 267 (6), 242 (14), 161 (27), 122 (100) *m/z*
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400 cm^{-1}, KBr pellet
	Peaks:	3451, 2943, 1665, 1622, 1371, 1295, 1238, 1157, 886 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Key Spectral data:	δ 0.93 (3H, s), 1.18 (3H, s), 1.24 (3H, s), 6.06 (1H, br s), 6.21 (1H, dd, *J* =1.9, 10.1 Hz), 7.05 (1H, d, *J* = 10.2 Hz) ppm
^{13}C NMR:	Instrument:	Bruker ARX-500
	Field strength:	126 MHz
	Solvent:	$CDCl_3$ (77.16 ppm)
	Spectral data:	δ 13.8, 18.5, 22.4, 23.2, 25.6, 31.2, 32.6, 33.1, 36.2, 38.6, 39.6, 43.4, 45.5, 49.7, 52.3, 81.2, 123.7, 127.3, 155.6, 168.9, 186.1 ppm
Melting point:		162-165 °C
Microanalysis:	Found:	C = 80.0%, H = 9.7% (April 2000)
	Calculated:	C = 80.0%, H = 9.4% (calculated for $C_{20}H_{28}O$)