Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

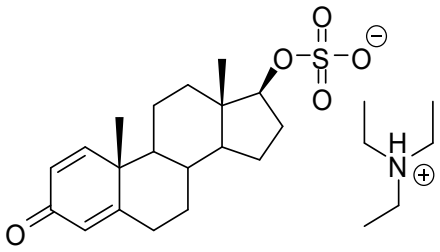
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D931: Boldenone sulfate, triethylamine salt

Report ID: D931.2024.02 (Bottled 231025)

Chemical Formula: $C_{25}H_{41}NO_5S$

Molecular Weight: 467.7 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
08-S-09	Not available	94.5 ± 0.6%

The uncertainty has been calculated according to ISO 33405 and is stated at the 95% confidence limit (k = 2).

Synonyms: 1,4-Androstadiene-3-one-17β-ol sulfate, triethylamine salt.

Expiration of certification: The property values are valid till 4 September 2029, 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 periodically 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: Off-white powder 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 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 HPLC with UV 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.
17 July 2026

This report supersedes any issued prior to 17 July 2026.

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:

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 HPLC with UV 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 LC-MS analysis, Q NMR analysis and elemental microanalysis.

HPLC:	Instrument:	Thermo Scientific UltiMate 3000 HPLC
	Column:	X-Bridge C-18, 5.0 µm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	A= 20 mM NH_4^+ HCO_2^- (aq) buffered to pH 10, B= Acetonitrile 0-18 min 22% B, 18-23 min 22-65% B, 23-25 min 65% B, 25-26 min 65-22% B, 26-35 min 22% B
	Flow rate:	1.0 mL/min, Gradient
	Detector:	RS Photodiode Array Detector operating at 248 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 98.4%, s = 0.02% (5 sub samples in duplicate, November 2023)
	Re-analysis:	Mean = 98.5%, s = 0.06% (5 sub samples in duplicate, September 2024)
HPLC:	Instrument:	Waters HPLC
	Column:	X-Bridge C-18, 5.0 µm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	A= 20 mM NH_4^+ HCO_2^- (aq) buffered to pH 10, B=Methanol 0-15 min 22% B, 15-25 min 22-90% B, 25-30 min 90% B, 30-31 min 90-22% B, 31-46 min 22% B
	Flow rate:	1.0 mL/min, Gradient
	Detector:	Waters Photodiode Array Detector operating at 248 nm
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 98.5%, s = 0.04% (6 sub samples in duplicate, June 2014)
	Re-analysis:	Mean = 98.7%, s = 0.02% (5 sub samples in duplicate, June 2017)
	Re-analysis:	Mean = 98.4%, s = 0.07% (5 sub samples in duplicate, May 2020)
QNMR:	Instrument:	Bruker DMX-600
	Field strength:	600 MHz
	Solvent:	DMSO-d_6 (2.50 ppm)
	Internal standard:	Dimethyl terephthalate
	Purity estimate:	Mean = 94.4%, s = 0.49% (5 sub samples in duplicate, April 2011)
Thermogravimetric analysis:		Non volatile residue 0.4% mass fraction (May 2008) Non volatile residue 0.2% mass fraction (October 2010)
Karl Fischer analysis:		Moisture content 2.6% mass fraction (September 2008) Moisture content 3.5% mass fraction (October 2009) Moisture content 3.8% mass fraction (October 2010) Moisture content 4.1% mass fraction (March 2014) Moisture content 4.5% mass fraction (June 2017) Moisture content 3.9% mass fraction (May 2020) Moisture content 4.1% mass fraction (November 2023) Moisture content 4.2% mass fraction (August 2024)

Spectroscopic and other characterisation data

LC-MS:	Instrument:	Waters 2695 (HPLC)/Micromass Quatro		
	Column:	X-Bridge C-18, 150 mm × 4.6 mm I.D. × 5 μm		
	Column temp:	40 °C		
	Solvent system:	20 mM NH_4^+ HCO_2^- (aq) buffered to pH 10, methanol		
	Flow rate:	0.2 mL/min		
	Sample prep:	2000 μg/g in MeOH/20 mM NH_4^+ HCO_2^- (aq) buffered to pH 10 (22:78)		
	Injection volume:	10 μL		
	Ionisation mode:	Electrospray negative ion		
	Capillary voltage:	3.0 kV	Cone voltage:	20 V
	Source temp:	130 ºC	Desolvation gas temperature:	350 ºC
	Cone gas flow rate:	27 L/hr	Desolvation gas flow rate:	748 L/hr
ESI-MS:	Instrument	Micromass Quatro Micro		
	Operation:	Negative ion mode, direct infusion at 5 μL/min		
	Ionisation:	ESI spray voltage at 3.2 kV negative ion		
	EM voltage:	500 V		
	Cone voltage:	20 V		
	Peak:	365 $(M\text{-}TEA^+)^-$ *m/z*		
TLC:	Conditions:	Kieselgel 60F_{254}. Dichloromethane / Methanol (9/1) Single, broad spot observed, R_f = 0.2 – 0.3. Visualisation with UV at 254 nm		
IR:	Instrument:	Biorad FTS300MX FT-IR		
	Range:	4000-400 cm^{-1}, KBr powder		
	Peaks:	3362, 2939, 2676, 2492, 1659, 1474, 1446, 1257, 1220, 1000, 804 cm^{-1}		
^{1}H NMR:	Instrument:	Bruker Gauss-400		
	Field strength:	400 MHz		
	Solvent:	MeOH-d_4 (3.30 ppm)		
	Spectral data:	δ 0.91 (3H, s), 0.98-1.09 (3H, m), 1.20 (1H, m), 1.28 (3H, s), 1.31 (9H, t, *J* = 7.3 Hz), 1.40 (1H, m), 1.59-1.84 (5H, m), 2.01 (2H, m), 2.16 (1H, m), 2.39 (1H, m), 2.58 (1H, m), 3.21 (6H, q, *J* = 7.3 Hz), 4.21 (1H, dd, *J* = 8.1, 8.9 Hz), 6.05 (1H, m), 6.20 (1H, dd, *J* = 1.9, 10.1 Hz), 7.29 (1H, d, *J* = 10.1 Hz) ppm		
^{13}C NMR:	Instrument:	Bruker Gauss-400		
	Field strength:	100 MHz		
	Solvent:	MeOH-d_4 (49.0 ppm)		
	Spectral data:	δ 9.2, 12.1, 19.1, 23.5, 24.4, 29.1, 33.8, 34.5, 36.6, 37.7, 44.1, 45.4, 47.9, 50.9, 54.2, 87.7, 124.0, 127.5, 159.6, 173.5, 188.6 ppm		
Melting point:		108-112 °C		
Microanalysis:	Found:	C = 62.3 %; H = 8.7 %; N = 3.0% (September 2008)		
	Calculated:	C = 64.2 %; H = 8.8 %; N = 3.0% (Calc. for $C_{25}H_{41}NO_5S$)		
	Calculated:	C = 62.3 %; H = 8.9 %; N = 2.9% (Calc. for $C_{25}H_{41}NO_5S$ containing 0.8 mole equivalents of moisture)		