Australian Government
Department of Industry, Science and Resources

National Measurement Institute

NATA
WORLD RECOGNISED ACCREDITATION

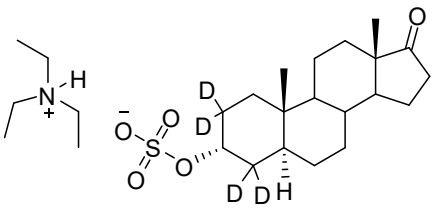
DEUTERATED INTERNAL STANDARD PRODUCT INFORMATION SHEET

NMIA D587: d_4-Androsterone sulfate (triethylammonium salt)

Report ID: D587.2026.01 (Ampouled 101014)

Chemical Formula: $C_{25}H_{41}D_4NO_5S$

Molecular Weight: 475.7 g/mol



Property value

Batch No.	CAS No.	Mass per ampoule
98-000501	**Not available**	**959 ± 23 μg**

IUPAC name: Triethylammonium (3α,5α)-17-Oxo(2,2, 4,4-$^2H_4$)androstan-3-yl sulfate

Expiration of certification: The property values are valid till 14 January 2031, 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 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 under an atmosphere of argon. The RM is intended for a single use to prepare a standard solution containing D587. The material was prepared by synthesis and certified for identity and purity by NMI Australia.

Intended use: The isotopic purity of this material is an estimate only. This material should be considered for use as an internal standard only.

Instructions for use: Open the ampoule and carefully rinse the interior at least three times with a suitable organic solvent (e.g. methanol). This will transfer approximately 959 ± 23 μg of anhydrous androsterone sulfate (triethylammomjium salt) (d_3, d_2, d_1 and d_0). The mass of analyte in each ampoule is calculated from the assigned purity of the bulk and the concentration of bulk material in a stock solution used to prepare the ampoules.

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: This material has demonstrated stability over a minimum period of three 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 HPLC with ELS detection 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 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.
10 February 2026.

This report supersedes any issued prior to 10 February 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:

HPLC:	Instrument:	Shimadzu Binary pump LC-20AB
	Column:	X-Bridge C-18, 5 μm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	Trifluoro acetic acid (0.05% v/v) was present in both aqueous and organic phases A = MilliQ water; B = Methanol 0-15 min 55% B; 15-17 min 55-80% B; 17-21 min 80%B; 21-23 min 80-55%B; 23-27 min 55%B.
	Flow rate:	1.0 mL/min
	Detector:	Shimadzu ELSD-LT II
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.8%, s = 0.3% (5 ampoules in duplicate, October 2010)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 ampoules in duplicate, September 2016)
	Re-analysis:	Mean = 99.9%, s = 0.01% (5 ampoules in duplicate, August 2021)
	Re-analysis:	Mean = 99.96%, s = 0.04% (5 ampoules in duplicate, January 2026)
HPLC:	Instrument:	Thermo Scientific UltiMate 3000
	Column:	X-Bridge C-18, 5 μm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	Trifluoro acetic acid (0.05% v/v) was present in both aqueous and organic phases A = MilliQ water; B = Methanol 0-15 min 55% B; 15-17 min 55-80% B; 17-21 min 80%B; 21-23 min 80-55%B; 23-27 min 55%B.
	Flow rate:	1.0 mL/min
	Detector:	Thermo Corona Veo RS CAD Detector
	Relative peak area of the main component:	
	Initial analysis:	Mean = 99.04%, s = 0.02% (5 ampoules in duplicate, January 2026)

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

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 elemental microanalysis.

The main component of this material is d_4-androsterone sulfate (triethylammonium salt). d_3-, d_2-, d_1- and d_0-Androsterone sulfate (triethylammonium salt) are also present. The stated chemical purity of the analyte represents the combined mass fractions of deuterated (d_4, d_3, d_2 and d_1) and d_0- androsterone sulfate (triethylammonium salt) in the material.

The isotopic purity of this material is an estimate only. This material should be considered for use as an internal standard only.

Isotopic Purity:	d_4 ≈ 94% [= $d_4/(d_4 + d_3 + d_2 + d_1 + d_0)$ x 100]	
	d_0 < 0.5% [= $d_0/(d_4 + d_3 + d_2 + d_1 + d_0)$ x 100]	
QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz
	Solvent:	$CDCl_3$ (7.26 ppm)
	Internal standard:	Dimethyl terephthalate (100.0% mass fraction)
	Initial analysis:	Mean (4.7 ppm) = 94.2%, s = 0.7% (5 sub samples, March 2017)
HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler
	Column:	X-Bridge C-18, 5 μm (4.6 mm x 150 mm)
	Column oven:	35 °C
	Mobile Phase:	Methanol/MilliQ water (55:45) Trifluoro acetic acid (0.05% v/v) was present in both aqueous and organic phases
	Flow rate:	1 mL/min
	Detector:	Waters ELSD
	Relative peak area of main component:	
	Initial analysis:	Mean = 99.8%, s = 0.01% (5 sub samples in duplicate, October 2010)
Thermogravimetric analysis:		Non-volatile residue 0.4% mass fraction (September 2016)
Karl Fischer analysis:		Moisture content 0.3-0.4% mass fraction (July 2006, September 2007 and September 2010), 2.6% mass fraction (March 2017)

Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Finnigan MAT TSQ 700
	Operation:	Negative ion mode, direct infusion
	Ionisation:	ESI probe at 4.5 kV
	Peak:	373 $(MSO3)^-$ *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Chloroform/methanol/water (70:20:2) Single spot observed, R_f = 0.30 (3 sub samples)
IR:	Instrument:	FT-IR, Biorad WIN FTS40
	Range:	4000-400 cm^{-1}, KBr pellet
	Peaks:	3500, 2712, 2361, 1738, 1269, 1198, 1014, 888, 659 cm^{-1}
^{1}H NMR:	Instrument:	Bruker DMX-500
	Field strength:	500 MHz
	Solvent:	d_6-DMSO (2.5 ppm)
	Key spectral data:	δ 0.74 (3H, s), 0.76 (3H, s), 1.16 (9H, t, *J* = 7.3 Hz), 3.09 (6H, q, *J* = 7.3 Hz), 4.25 (1H, s) ppm
	As a result of successful deuteration, no absorptions or couplings observed due to hydrogen at 2- or 4-position.	
^{13}C NMR:	Instrument:	Bruker DMX-500
	Field strength:	126 MHz
	Solvent:	d_6-DMSO (39.5 ppm)
	Spectral data:	δ 9.0, 11.6, 13.8, 20.0, 21.7, 28.2, 30.9, 31.8, 32.6, 34.9, 35.7, 39.4, 40.2, 46.2, 47.5, 51.1, 54.4, 71.1, 220.2 ppm As a result of successful deuteration, signals due to C-2 and C-4 are not observed above baseline noise.
Microanalysis:	Found:	C = 62.8%; H/D = 10.3%; N = 3.0% (June 1999)
	Calculated:	C = 63.1%; H/D = 10.4%; N = 2.9% (Calculated for $C_{25}H_{41}D_4NO_5S$)