



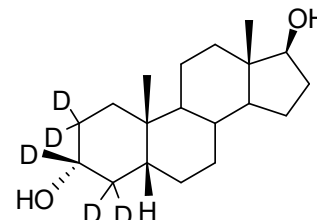
DEUTERATED INTERNAL STANDARD PRODUCT INFORMATION SHEET

NMIA D580b: d₅-5 β -Androstane-3 α , 17 β -diol

Report ID: D580b.2026.01 (Ampouled 251001)

Chemical Formula: C₁₉H₂₇D₅O₂

Molecular Weight: 297.4 g/mol



Property value

Batch No.	CAS No.	Mass per ampoule
21-S-02	361432-68-0	989 ± 18 µg

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

IUPAC name: (2,2,3 β ,4,4-²H₅)-(3 α , 5 β , 17 β)-Androstane-3,17-diol.

Expiration of certification: The property values are valid till 30 September 2029, 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 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 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: White solid prepared by synthesis and certified for identity and purity by NMI Australia. The analyte is supplied as a dried aliquot in a sealed ampoule under an atmosphere of argon, intended for single use to prepare a standard solution. The main component of this material is d₅-5 β -androstane-3 α ,17 β -diol. d₄-, d₃-, d₂-, d₁- and d₀-5 β -Androstane-3 α ,17 β -diol are also present. The stated mass of the analyte per ampoule represents the approximate combined masses of deuterated (d₅, d₄, d₃, d₂ and d₁) and d₀-5 β -androstane-3 α ,17 β -diol in the material.

Intended use: The isotopic purity of this material is an estimate only. This material should be considered for use as an internal standard only and is not intended for use as a calibrator. The material does not have certified reference material status as metrological traceability of the stated purity value to the SI unit for mass (kg) has not been established.

Instructions for use: Open the ampoule and carefully rinse the interior at least three times, each time with a minimum of 0.5 mL of a suitable organic solvent (e.g. 2-propanol). This process will ensure the transfer of the stated mass per ampoule of anhydrous 5 β -androstane-3 α ,17 β -diol (d₅, d₄, d₃, d₂, d₁ and d₀). The mass of analyte contained in each ampoule has been determined based on the assigned purity of the bulk material and the concentration of that bulk material in the stock solution used during ampoule preparation.

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.

Stability: In the absence of long term stability data the measurement uncertainty at the 95% coverage interval has been expanded to accommodate any potential change in the property value. The stability component has been estimated from stability trials conducted on similar materials by NMI Australia over the last ten 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 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 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.
7 October 2026.

This report supersedes any issued prior to 07 October 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:

GC-FID: Instrument: Agilent 8890
 Column: PH-1ms, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 170 °C (1 min), 10 °C/min to 200 °C (5 min), 20 °C/min to 300 °C (5 min)
 Injector: 250 °C
 Detector Temp: 320 °C
 Carrier: Helium
 Split ratio: 20/1
 Relative peak area of the main component:
 Initial analysis: Mean = 99.2%, s = 0.06% (7 ampoules in duplicate, October 2025)
 Re-analysis: Mean = 99.5%, s = 0.03% (5 ampoules in duplicate, September 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 indicative purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis, and ¹H NMR spectroscopy. The purity value is calculated as per Equation 1.

$$\text{Purity} = (100 \% - I_{\text{ORG}}) \times (100 \% - I_{\text{VOL}} - I_{\text{NVR}}) \quad \text{Equation 1}$$

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

Supporting evidence is provided by elemental microanalysis.

The main component of this material is d₅-5β-androstane-3α,17β-diol. d₄-, d₃-, d₂-, d₁- and d₀- 5β-androstane-3α,17β-diol are also present. The stated chemical purity of the analyte represents the combined mass fractions of deuterated (d₄, d₃, d₂ and d₁) and d₀- 5β-androstane-3α,17β-diol 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: $d_4 \approx 88\% [= d_4/(d_4 + d_3 + d_2 + d_1 + d_0) \times 100]$
 $d_0 < 0.1\% [= d_0/(d_4 + d_3 + d_2 + d_1 + d_0) \times 100]$

GC-FID: Instrument: Agilent 8890
 Column: HP-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
 Program: 170 °C (1 min), 10 °C/min to 200 °C (5 min), 20 °C/min to 300 °C (3 min)
 Injector: 250 °C
 Detector Temp: 320 °C
 Carrier: Helium
 Split ratio: 20/1
 Relative peak area of the main component:
 Initial analysis: Mean = 99.5%, s = 0.02% (10 sub samples in duplicate, June 2021)

Karl Fischer analysis: Moisture content 0.4% mass fraction (July 2021)

Thermogravimetric analysis: Volatiles content 0.4% and non-volatile residue < 0.2% mass fraction (June 2021)

Spectroscopic and other characterisation data

GC-MS:	Parent compound:	
	Instrument:	Agilent 6890/5973
	Column:	DB-5MS, 30 m x 0.25 mm I.D. x 0.25 μ m
	Program:	180 $^{\circ}$ C (1 min), 30 $^{\circ}$ C/min to 250 $^{\circ}$ C (10 min), 30 $^{\circ}$ C/min to 300 $^{\circ}$ C (3 min)
	Injector:	250 $^{\circ}$ C
	Split ratio:	20/1
	Transfer line temp:	280 $^{\circ}$ C
	Carrier:	Helium, 1.0 mL/min
	Scan range:	50-550 m/z
	<i>Bis</i> -TMS derivative:	
	Instrument:	HP 6890/5973
	Column:	DB-5MS, 30 m x 0.25 mm I.D. x 0.25 μ m
	Program:	170 $^{\circ}$ C (0.5 min), 3 $^{\circ}$ C/min to 234 $^{\circ}$ C, 10 $^{\circ}$ C/min to 265 $^{\circ}$ C (3 min)
	Injector:	250 $^{\circ}$ C
	Split ratio:	20/1
	Transfer line temp:	280 $^{\circ}$ C
	Carrier:	Helium
	Scan range:	50-550 m/z
	The retention times of the parent compound and <i>bis</i> -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 (8.9 min):	279 (64), 261 (47), 246 (37), 235 (33), 220 (100) m/z
	<i>Bis</i> -TMS (9.3 min):	441 (M^+ , 4), 351 (16), 261 (65), 220 (38), 129 (78), 73 (100) m/z
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Chloroform/methanol (9:1) Single spot observed, R_f = 0.7. Visualisation with UV at 254 nm.
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 cm^{-1} , neat
	Peaks:	3302, 2918, 2859, 2195, 2105, 1444, 1376, 1346, 1312, 1197, 1104, 1056, 1030, 944, 738 cm^{-1}
^1H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	MeOH- d_4 (3.31 ppm)
	Spectral data:	δ 0.72 (3H, s), 0.96 (3H, s), 0.99-1.16 (4H, m), 1.20-1.32 (3H, m), 1.38-1.50 (6H, m), 1.59 (1H, m), 1.79-2.01 (4H, m), 3.58 (1H, t, J = 8.7 Hz), ppm Ethyl acetate estimated at 0.3% mass fraction was observed in the ^1H NMR
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	MeOH- d_4 (49 ppm)
	Spectral data:	δ 11.6, 21.5, 23.9, 24.3, 27.3, 28.2, 30.7, 35.7, 36.4, 37.3, 38.2, 42.1, 43.4, 44.2, 52.4, 82.6 ppm
Melting point:		234-235 $^{\circ}$ C
Microanalysis:	Found:	C = 76.7%; H = 11.0% (July, 2021)
	Calculated:	C = 76.7%; H = 11.0% (Calculated for C ₁₉ H ₂₇ D ₅ O ₂)