



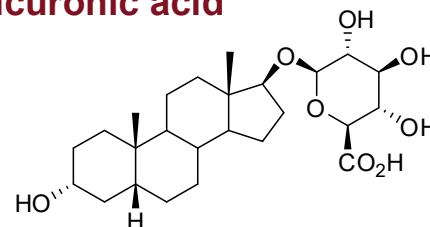
# CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

## NMIA S005b: 5 $\beta$ -Androstane-3 $\alpha$ , 17 $\beta$ -diol-17-O- $\beta$ -Glucuronic acid

Report ID: S005b.2026.01

Chemical Formula: C<sub>25</sub>H<sub>40</sub>O<sub>8</sub>

Molecular Weight: 468.6 g/mol



### Certified value

Batch No.	CAS No.	Purity (mass fraction)
23-S-06	7776-46-7	95.0 ± 2.1%

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

**IUPAC name:** (3 $\alpha$ ,5 $\beta$ ,17 $\beta$ )-Androstan-3,17-diol-17-yl  $\beta$ -D-glucopyranosiduronic acid

**Expiration of certification:** The property values are valid till 20 March 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 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 on an annual basis 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 powder prepared by synthesis and certified for identity and purity by NMI Australia. Packaged in amber glass bottles with a septum and crimped aluminium 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 °C 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 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:** 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 measurement uncertainty at the 95% confidence interval also includes a stability component determined from accelerated stability trials conducted at 40 °C and 75% humidity for a 14 day period.

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 charged aerosol 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.  
9 April 2026

This report supersedes any issued prior to 09 April 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.

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## 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 charged aerosol detection, thermogravimetric analysis, Karl Fischer analysis, and <sup>1</sup>H NMR spectroscopy. The purity value is calculated as per Equation 1.

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

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

Supporting evidence is provided by elemental microanalysis.

HPLC: Instrument: Thermo Scientific UltiMate 3000  
 Column: Avantor ACE Excel 5 SuperC18, 5 μm (4.6 mm x 150 mm)  
 Column oven: 40 °C  
 Mobile Phase: Milli Q water with 0.5% HCO<sub>2</sub>H / methanol (35:65 v/v)  
 Flow rate: 1.0 mL/min  
 Detector: CAD Detector  
 Relative mass fraction of the main component:  
 Initial analysis: Mean = 98.7%, s = 0.05% (10 sub samples in duplicate, March 2025)  
 Re- analysis: Mean = 98.3%, s = 0.03% (5 sub samples in duplicate, March 2026)

HPLC: Instrument: Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler  
 Column: Avantor ACE Excel 5 SuperC18, 5 μm (4.6 mm x 150 mm)  
 Column oven: 40 °C  
 Mobile Phase: Milli Q water with 0.5% HCO<sub>2</sub>H / methanol (35:65 v/v)  
 Flow rate: 1.0 mL/min  
 Detector: Shimadzu ELSD-LT II  
 Relative mass fraction of the main component:  
 Initial analysis: Mean = 99.9%, s = 0.01% (10 sub samples in duplicate, March 2025)

HPLC: Instrument: Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler  
 Column: Avantor ACE Excel 5 SuperC18, 5 μm (4.6 mm x 150 mm)  
 Column oven: 40 °C  
 Mobile Phase: Milli Q water with 0.5% HCO<sub>2</sub>H / methanol (35:65 v/v)  
 Flow rate: 1.0 mL/min  
 Detector: Shimadzu ELSD-LT III  
 Relative mass fraction of the main component:  
 Initial analysis: Mean = 99.98%, s = 0.001% (5 sub samples in duplicate, March 2026)

Karl Fischer analysis: Moisture content 3.9% mass fraction (April 2025)  
 Moisture content 3.7% mass fraction (March 2026)

Thermogravimetric analysis: Volatiles content 3.6% and non-volatile residue 0.4% mass fraction (March and May 2025)

### Spectroscopic and other characterisation data

ESI-MS:	Instrument:	Shimadzu
	Operation:	Negative ion mode, direct infusion at 10 $\mu$ L/min
	Interface voltage:	ESI spray voltage at 4.0 kV negative ion
	Peak:	467 (M-H) $m/z$
IR:	Instrument:	Bruker Alpha Platinum ATR
	Range:	4000-400 $\text{cm}^{-1}$ , neat
	Peaks:	3465, 2964, 2929, 2905, 2868, 1684, 1449, 1375, 1259, 1232, 1182, 1094, 1019, 943, 687 $\text{cm}^{-1}$
$^1\text{H}$ NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	MeOH- $d_4$ (3.31 ppm)
	Spectral data:	$\delta$ 0.82 (3H, s), 0.95 (3H, s), 0.95-1.48 (17H, m), 1.54-1.67 (3H, m), 1.72-2.04 (5H, m), 3.21 (1H, dd, $J$ = 7.8, 9.1 Hz), 3.36 (1H, t, $J$ = 9.2 Hz), 3.51 (1H, t, $J$ = 9.5 Hz), 3.54 (1H, m), 3.69 (1H, t, $J$ = 8.6 Hz), 3.74 (1H, d, $J$ = 9.8 Hz), 4.38 (1H, d, $J$ = 7.8 Hz) ppm
$^{13}\text{C}$ NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	MeOH- $d_4$ (49 ppm)
	Spectral data:	$\delta$ 12.1, 21.5, 23.9, 24.3, 27.2, 28.2, 29.9, 31.2, 35.8, 36.5, 37.1, 37.1, 38.9, 42.0, 43.6, 44.4, 52.2, 72.4, 73.2, 75.0, 76.6, 77.6, 90.5, 105.1, 172.6 ppm
Microanalysis:	Found:	C = 61.7%; H = 8.9% (May 2025)
	Calculated:	C = 64.1%; H = 8.6% (Calculated for $\text{C}_{25}\text{H}_{40}\text{O}_8$ )
	Calculated:	C = 61.7%; H = 8.7% (Calculated for $\text{C}_{25}\text{H}_{40}\text{O}_8 \cdot \text{H}_2\text{O}$ )