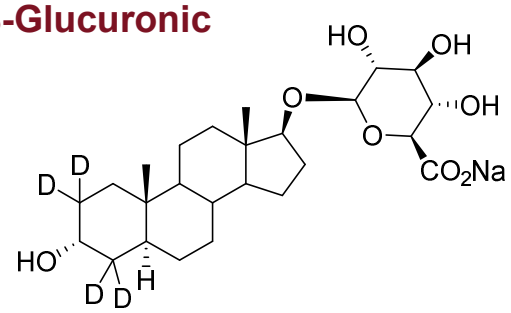
DEUTERATED INTERNAL STANDARD PRODUCT INFORMATION SHEET

NMIA S062: d4-5α-Androstane-3α, 17β-diol-17-O-β-Glucuronic acid, sodium salt



Report ID: S062.2026.01

Chemical Formula: $C_{25}H_{35}D_4O_8Na$

Molecular Weight: 494.6 g/mol

Property value

Batch No.	CAS No.	Purity estimate
25-S-03	N/A	73.5 ± 4.2%

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

IUPAC name: N/A.

Expiration of certification: The property values are valid till 13 Jan 2029, three years from the date of 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: 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: 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: 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.

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 HPLC with charge 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.
3 February 2026

NATA Accreditation No. 198 / Corporate Site No. 14214.

Characterisation Report:

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 charged aerosol detection, 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 elemental microanalysis. The main component of this material is d_4-5α-androstan-3α,17β-diol-17-O-β-glucuronide sodium salt. d_3-, d_2-, d_1- and d_0-5α-androstan-3α,17β-diol-17-O-β-glucuronide sodium 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_0-5α-androstan-3α,17β-diol-17-O-β-glucuronide sodium 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: $d_4 \approx 88\%$ [= $d_4/(d_4 + d_3 + d_2 + d_1 + d_0)$ x 100]

$d_0 < 0.2\%$ [= $d_0/(d_4 + d_3 + d_2 + d_1 + d_0)$ x 100]

HPLC:	Instrument:	Thermo Scientific Vanquish pump
	Column:	X-bridge C-18, 5 μm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	A = 0.5 percent formic acid in MilliQ water; B = Acetonitrile 0-5 min 30% B; 5-15 min 30-50% B; 15-16 min 50%B; 16-17 min 50-30%B; 17-20 min 30%B
	Flow rate:	1.0 mL/min
	Detector:	Vanquish Detector
	Relative peak area of the main component:	
	Initial analysis:	Mean = 74.0%, s = 0.3% (10 sub samples in duplicate, January 2026)
HPLC:	Instrument:	Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
	Column:	X-bridge C-18, 5 μm (4.6 mm x 150 mm)
	Column oven:	40 °C
	Mobile Phase:	A = 0.5 percent formic acid in MilliQ water; B = Acetonitrile 0-5 min 30% B; 5-15 min 30-50% B; 15-16 min 50%B; 16-17 min 50-30%B; 17-20 min 30%B
	Flow rate:	1.0 mL/min
	Detector:	Shimadzu ELSD-LT II
	Relative peak area of the main component:	
	Initial analysis:	Mean = 91.6%, s = 0.1% (10 sub samples in duplicate, January 2026)
Karl Fischer analysis:		Moisture content 12.1% mass fraction (December 2025)

Spectroscopic and other characterisation data

LC-MS:	Instrument:	Shimadzu LC-TQ-MS 8045
	Column:	ACE Excel 5 Super C18, 150 mm × 4.6 mm I.D. × 5 μm
	Column temp:	40 °C
	Solvent system:	methanol/0.1% formic acid (70 :30 v/v)
	Flow rate:	1.0 mL/min
	Sample prep:	1 μg/g in methanol
	Injection volume:	10 μL
	Ionisation mode:	Electrospray negative ion
	Interface voltage:	3.0 kV
	The retention time of d_4-5α-androstan-3α,17β-diol-17-O-β-glucuronide sodium salt is reported with the major peak in the mass spectrum. The latter is reported as a mass/charge ratio.	
	6.2 min:	471.0 ($M\text{-}H^+$) *m/z*
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	MeOH-d_4 (3.31 ppm)
	Spectral data:	δ 0.82 (3H, s), 0.83 (3H, s), 0.64-1.13 (3H, m), 1.18-1.45 (7H, m), 1.52-1.72 (5H, m), 1.98 (1H, m), 2.08 (1H, m), 3.19 (1H, t, *J* = 9.0 Hz), 3.38 (1H, t, *J* = 9.0 Hz), 3.42 (1H, t, *J* = 8.5 Hz), 3.50 (1H, d, *J* = 9.5 Hz), 3.82 (1H, t, *J* = 8.5 Hz), 3.93 (1H, s), 4.35 (1H, d, *J* = 8.0 Hz) ppm
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	MeOH-d_4 (49 ppm)
	Spectral data:	δ 11.7, 12.1, 21.5, 24.2, 29.5, 29.7, 32.9, 33.3, 36.8, 37.1, 39.0, 40.2, 44.4, 52.4, 56.0, 67.0, 73.8, 75.3, 76.4, 78.0, 89.4, 104.5, 177.0 ppm
Microanalysis:	Found:	C = 52.6%; H = 7.9%; (December, 2025)
	Calculated:	C = 60.7%; H = 8.7%; (Calculated for $C_{25}H_{35}D_4O_8Na$)