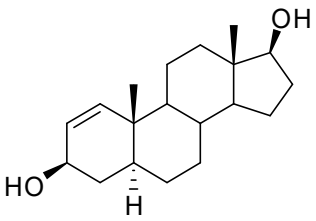
# REFERENCE MATERIAL PRODUCT INFORMATION SHEET

## NMIA D871b: 5α-Androst-1-ene-3β,17β-diol




Report ID: D871b.2021.03 (Ampouled 130805)

Chemical Formula: $C_{19}H_{30}O_2$

Molecular Weight: 290.4 g/mol

## Property value

| Batch No. | CAS No. | Mass per ampoule |
|---|---|---|
| **12-S-02** | **5323-27-3** | **911 ± 24 μg** |

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

**IUPAC name:** (3β,5α,17β)-Androst-1-ene-3,17-diol.

**Expiration of certification:** The property values are valid till 3 December 2026, 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 ampoules that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials. In the event a product fails the stability trial, notification will be sent to all impacted customers.

**Description:** The compound is supplied as a dried aliquot in a sealed ampoule under an atmosphere of argon. The CRM is intended for a single use to prepare a standard solution containing D871b. This material was prepared by synthesis, and certified for identity and purity by NMIA.

**Intended use:** This reference material is recommended for qualitative analysis 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 with a suitable organic solvent (e.g. chloroform). This will transfer approximately 911 μg of anhydrous 5α-androst-1-ene-3β,17β-diol. 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 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 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 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 December 2025

This report supersedes any issued prior to 10 December 2025.

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 6890 or 8890 |
| Column: | HP-1 or HP5, 30 m × 0.32 mm I.D. × 0.25 μm |
| Program: | 180 °C (1 min), 5 °C/min to 210 °C (14min), 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 = 96.4%, s = 0.08% (7 ampoules in duplicate, August 2013) |
| Re-analysis: | Mean = 96.4%, s = 0.09% (5 ampoules in duplicate, July 2014) |
| Re-analysis: | Mean = 96.3%, s = 0.06% (5 ampoules in duplicate, May 2017) |
| Re-analysis: | Mean = 96.1%, s = 0.04% (5 ampoules in duplicate, December 2021) |

**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 certified 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 headspace GC-MS analysis of occluded solvents and elemental microanalysis.

**Warning:** **The parent compound and *bis*-TMS derivatise are sensitive to the quality of the silanised glass liner when injected at elevated temperature (~ 160-250 ºC) into a GC instrument.**

GC-FID:

| | |
|---|---|
| Instrument: | Varian CP-3800 |
| Column: | HP-5, 30 m × 0.32 mm I.D. × 0.25 μm |
| Program: | 180 °C (1 min), 30 °C/min to 250 °C (10 min), 30 °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 = 96.6%, s = 0.1% (10 sub samples in duplicate, September 2012)

GC-FID:

| | |
|---|---|
| Instrument: | Agilent 6890 |
| Column: | HP-1, 30 m × 0.32 mm I.D. × 0.25 μm |
| Program: | 180 °C (1 min), 10 °C/min to 220 °C (10 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 = 97.0%, s = 0.1% (10 sub samples in duplicate, September 2012)

Thermogravimetric analysis: Volatile content 5.3% and non volatile residue < 0.2% mass fraction (September 2012)

Karl Fischer analysis: Moisture content 3.3% mass fraction (September 2012)

## Spectroscopic and other characterisation data

| | | |
|---|---|---|
| GC-MS: | Parent compound: | |
| | Instrument: | Agilent 6890/5973 |
| | Column: | TG1-MS, 30 m x 0.25 mm I.D. x 0.25 μm |
| | Program: | 180 °C (1 min), 10 °C/min to 300 °C (2 min) |
| | Injector: | 250 °C |
| | Transfer line temp: | 280 °C |
| | Carrier: | Helium, 1.0 mL/min |
| | Split ratio: | 20/1 |
| | *Bis*-TMS derivative: | |
| | Instrument: | Agilent 6890/5973 |
| | Column: | TG1-MS, 30 m x 0.25 mm I.D. x 0.25 μm |
| | Program: | 180 °C (1 min), 10 °C/min to 300 °C (2 min) |
| | Injector: | 250 °C |
| | Transfer line temp: | 280 °C |
| | Carrier: | Helium, 1.0 mL/min |
| | Split ratio: | 20/1 |
| | The retention times of the parent compound and *bis*-TMS derivative are reported along 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 (10.2 min): | 290 ($M^+$, 39), 272 (29), 220 (69), 202 (37), 187 (35), 161 (45), 147 (36), 135 (25), 118 (36), 105 (72), 91 (100), 79 (60), 55 (42) *m/z* |
| | *Bis*-TMS (11.4 min): | 434 ($M^+$, 51), 405 (25), 202 (19), 143 (100), 129 (39), 127 (34), 105 (24), 75 (75), 73 (90) *m/z* |
| HS-GC-MS: | Instrument: | Agilent 6890/5973/G1888 |
| | Column: | DB-624, 30 m x 0.25 mm I.D. x 1.4 μm |
| | Program: | 50 °C (5 min), 7 °C/min to 120 °C, 15 °C /min to 220 °C (8.3 min) |
| | Injector: | 150 °C |
| | Transfer line temp: | 280 °C |
| | Carrier: | Helium, 1.2 mL/min |
| | Split ratio: | 50/1 |
| | Solvents detected: | Ethyl acetate, hexane, chloroform |
| TLC: | Conditions: | Kieselgel 60$F_{254}$. Chloroform/ethyl acetate (2/1)<br>Single spot observed, $R_f$ = 0.4. Visualisation with vanillin |
| IR: | Instrument: | Biorad FTS3000MX FT-IR |
| | Range: | 4000-400 $cm^{-1}$, KBr powder |
| | Peaks: | 3463, 3392, 3221, 3021, 2929, 2845, 1645, 1446, 1324, 1126, 1067, 1026, 976, 871, 753, 605 $cm^{-1}$ |
| $^1$H NMR: | Instrument: | Bruker Avance-400 |
| | Field strength: | 400 MHz |
| | Solvent: | $CDCl_3$ (7.26 ppm) |
| | Spectral data: | δ 0.74 (3H, s), 0.79-1.08 (4H, m), 0.91 (3H, s), 1.19-1.61 (11H, m), 1.67-1.84 (4H, m), 2.09 (1H, m), 3.62 91H, bm), 4.29 (1H, bs), 5.48 (1H, ddd, *J* = 1.7, 1.7, 10.2 Hz), 5.91 (1H, dd, *J* = 1.8, 10.2 Hz) ppm |
| | Ethyl acetate estimated at 0.5% mass fraction was observed in the $^1$H NMR, while chloroform and hexane could not be quantified due to overlapping peaks. | |
| $^{13}$C NMR: | Instrument: | Bruker Avance-400 |
| | Field strength: | 101 MHz |
| | Solvent: | $CDCl_3$ (77.0 ppm) |
| | Spectral data: | δ 11.2, 15.7, 20.8, 23.3, 27.9, 30.5, 31.4, 35.6, 35.8, 36.6, 38.1, 43.1, 43.5, 51.0, 51.5, 68.8, 81.8, 128.7, 137.9 ppm |
| Melting point: | | 159-162 °C |
| Microanalysis: | Found: | C = 76.8%; H = 10.6% (September, 2012) |
| | Calculated: | C = 78.6%; H = 10.4% (Calculated for $C_{19}H_{30}O_2$) |