## National Measurement Institute

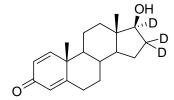


# DEUTERATED INTERNAL STANDARD PRODUCT INFORMATION SHEET

## NMIA D581c: d3-Boldenone

Report ID: D581c.2025.01 (Ampouled 250403)

Chemical Formula: C<sub>19</sub>H<sub>23</sub>D<sub>3</sub>O<sub>2</sub> Molecular Weight: 289.4 g/mol



## **Property value**

Batch No.	CAS No.	Mass per ampoule
23-S-01	1224710-35-3	991 ± 20 μg

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

**IUPAC name:** (17β)-17-Hydroxy(16,16,17-<sup>2</sup>H<sub>3</sub>) androst-1,4-dien-3-one.

**Expiration of certification:** The property values are valid till 4 April 2028, 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 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 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 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  $d_3$ -boldenone,  $d_2$ -,  $d_1$ - and  $d_0$ -boldenone are also present. The stated mass of the analyte per ampoule represents the approximate combined masses of deuterated ( $d_3$ ,  $d_2$  and  $d_1$ ) and  $d_0$ -boldenone 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 with a suitable organic solvent (e.g. chloroform). This will transfer approximately 991  $\pm$  20  $\mu$ g of anhydrous boldenone (d<sub>3</sub>, d<sub>2</sub>, d<sub>1</sub> and d<sub>0</sub>). 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.

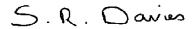
Recommended storage: When not in use, this material should be stored at or below 4 °C in a closed container in a dry, dark area

**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.



Dr Stephen R. Davies, Team Leader, Chemical Reference Materials, NMI. 14 May 2025.

This report supersedes any issued prior to 14 May 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 8890

Column: HP-5, 30 m  $\times$  0.32 mm l.D.  $\times$  0.25  $\mu$ m

Program: 180 °C (1 min), 30 °C/min to 250 °C (10 min), 30 °C/min to 300 °C (3 min)

Injector: 200 °C

Detector Temp: 320 °C

Carrier: Helium

Split ratio: 20/1

Relative peak area of the main component:

Initial analysis: Mean = 98.9%, s = 0.02% (7 ampoules in duplicate, April 2025)

#### 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 <sup>1</sup>H NMR spectroscopy. The purity value is calculated as per Equation 1.

Equation 1

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

I<sub>ORG</sub> = Organic impurities of related structure, I<sub>VOL</sub> = volatile impurities, I<sub>NVR</sub> = non-volatile residue.

Supporting evidence is provided by elemental microanalysis.

The main component of this material is  $d_3$ -boldenone.  $d_2$ -,  $d_1$ - and  $d_0$ -Boldenone are also present. The stated chemical purity of the analyte represents the combined mass fractions of deuterated ( $d_3$ ,  $d_2$  and  $d_1$ ) and  $d_0$ -boldeneone 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:  $d_3 \approx 91\% [= d_3/(d_3 + d_2 + d_1 + d_0) \times 100]$ 

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

GC-FID: Instrument: Agilent 8890

Column: HP-1, 30 m  $\times$  0.32 mm I.D.  $\times$  0.25  $\mu$ m

Program: 190 °C (1 min), 15 °C/min to 270 °C (3 min), 30 °C/min to 300 °C (3 min)

Injector: 200 °C

Detector Temp: 320 °C

Carrier: Helium

Split ratio: 20/1

Relative peak area of the main component:

Initial analysis: Mean = 98.9%, s = 0.02% (10 sub samples in duplicate, March 2025)

GC-FID: Instrument: Agilent 8890

Column: HP-5, 30 m  $\times$  0.32 mm I.D.  $\times$  0.25  $\mu$ m

Program: 190 °C (1 min), 15 °C/min to 270 °C (3 min), 30 °C/min to 300 °C (3 min)

Injector: 200 °C

Detector Temp: 320 °C

Carrier: Helium

Split ratio: 20/1

Relative peak area of the main component:

Initial analysis: Mean = 99.0%, s = 0.02% (10 sub samples in duplicate, March 2025)

Karl Fischer analysis: Moisture content ≤ 0.1% mass fraction (March 2025)

Thermogravimetric analysis: Volatiles content 0.2% and non-volatile residue < 0.2% mass fraction (March 2025)

### Spectroscopic and other characterisation data

GC-MS: Instrument: Agilent 8890/5977B

Column: HP-5MS, 30 m x 0.25 mm I.D. x 0.25  $\mu$ m

Program: 190 °C (1 min), 15 °C/min to 270 °C (3 min), 30 °C/min to 300 °C (3 min)

Injector: 250 °C Split ratio: 20/1 Transfer line temp: 280 °C

Carrier: Helium, 1.0 mL/min

Scan range: 50-550 *m/z* 

The retention time of d₃-boldenone is 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 (10.4 min): 289 (M+, 1), 122 (100), 108 (8), 107 (10), 91 (33), 79 (9), 77 (5) m/z

TLC: Conditions: Kieselgel 60F<sub>254</sub>. Hexane/ethyl acetate (1:1)

Single spot observed,  $R_f = 0.3$ . Visualisation with UV at 254 nm.

IR: Instrument: Biorad FTS300MX FT-IR Range: 4000-400cm<sup>-1</sup>, KBr pellet

Peaks: 3479, 2939, 2873, 2851, 2216, 2158, 2119, 1661, 1619, 1599, 1453, 1378, 1299,

1240, 1188, 1166, 1040, 958, 885, 821 cm<sup>-1</sup>

<sup>1</sup>H NMR: Instrument: Bruker Avance III-500

Field strength: 500 MHz Solvent: CDCl<sub>3</sub> (7.26 ppm)

Spectral data: δ 0.81 (3H, s), 0.89-1.14 (4H, m), 1.24 (3H, s), 1.31 (1H, t, *J* = 12 Hz), 1.55-1.81 (4H,

m), 1.87 (1H, ddd, J = 2.8, 4.0, 13.0 Hz), 1.95 (1H, m), 2.36 (1H, ddd, J = 2.5, 4.4, 13.0 Hz), 2.47 (1H, tdd, J = 1.6, 5.3, 13 Hz), 6.08 (1H, t, J = 1.7 Hz), 6.23 (1H, dd, J = 1.9, 10

Hz), 7.06 (1H, d, J = 10 Hz) ppm.

Dichloromethane and methanol were quantified at 0.14% and 0.03%, respectively.

<sup>13</sup>C NMR: Instrument: Bruker Avance III-500

Field strength: 126 MHz

Solvent: CDCl<sub>3</sub> (77.16 ppm)

Spectral data: δ 11.3, 18.8, 22.7, 23.4, 32.9, 33.3, 35.7, 36.4, 43.1, 43.8, 50.2, 52.7, 123.9, 127.5,

156.3, 169.6, 186.6 ppm.

Melting point: 172-174 °C

Microanalysis: Found: C = 79.2%; H = 9.2% (April 2025)

Calculated: C = 78.9%; H = 9.2% (Calculated for  $C_{19}H_{23}D_3O_2$ )