



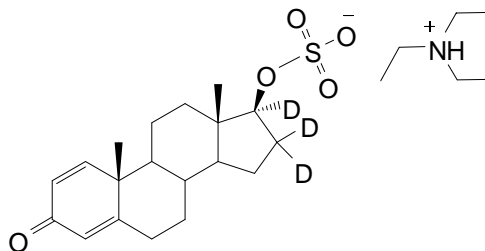
DEUTERATED INTERNAL STANDARD PRODUCT INFORMATION SHEET

NMIA S013: d₃-Boldenone sulfate triethylamine salt

Report ID: S013.2019.01 (Ampouled 121015)

Chemical Formula: C₂₅H₃₈D₃NO₅ S

Molecular Weight: 470.7 g/mol



Property value

| Batch No. | CAS No. | Mass per ampoule |
|-----------|---------------|------------------|
| 11-S-12 | Not available | 891 µg |

Synonyms: 17β-(Sulfoxy)-androsta-1,4-dien-3-one-16,16,17-d₃ triethylamine salt.

Expiration of certification: The property values are valid till 9 July 2024, 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 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.

Description: The compound is supplied as a dried aliquot in a sealed ampoule and is intended for a single use to prepare a standard solution containing S013. The material was prepared by synthesis, and certified for identity and purity by NMIA.

Intended use: The isotopic purity of this material is an estimate only. This material should be considered for use as an internal standard only.

Instructions for use: Open the ampoule and carefully rinse the interior at least three times with a suitable organic solvent (e.g. methanol). This will transfer approximately 891 µg of anhydrous boldenone sulfate triethylamine salt (d₃, d₂, d₁ and d₀).

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: This material has demonstrated stability over a minimum period of three 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 UV detection 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.
2 August 2019.

This report supersedes any issued prior to 2 August, 2019.

NATA logo notice: Accredited for compliance with ISO Guide 17034. Accreditation No. 198 / Corporate Site No. 20844. The results of the tests, calibrations and/or measurements included in this document are traceable to Australian/national standards.

Legal notice: Terms and Conditions associated with the provision of this reference material can be found on the NMIA website.

Characterisation Report:

| | | |
|-------|---|---|
| HPLC: | Instrument: | Waters Model 1525 Binary pump, 717 plus autosampler or Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler |
| | Column: | X-Bridge C-18, 5 µm (4.6 mm × 150 mm) |
| | Column oven: | 40 °C |
| | Mobile Phase: | MilliQ water/Methanol A = MilliQ water; B = Methanol 0.05%TFA was present in both aqueous and organic phase 0-14 min 44% B; 14-18 min 44-80% B; 18-22 min 80% B; 22-23 min 80-44% B, 23-30 min 44 % B |
| | Flow rate: | 1.0 mL/min |
| | Detector: | Shimadzu SPD-M20A or Waters 2998 PDA operating at Max plot. |
| | Relative peak area of the main component: | |
| | Initial analysis: | Mean = 93.0%, s = 0.18% (7 ampoules in duplicate, October 2012) |
| | Re-analysis: | Mean = 93.2%, s = 0.04% (5 ampoules in duplicate, September 2013) |
| | Re-analysis: | Mean = 93.2%, s = 0.04% (5 ampoules in duplicate, August 2016) |
| | Re-analysis: | Mean = 93.1%, s = 0.04% (5 ampoules in duplicate, July 2019) |

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 purity value was obtained by mass balance from a combination of traditional analytical techniques, including HPLC with UV, 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 qualitative elemental microanalysis.

The main component of this material is d₃-boldenone sulfate triethylamine salt. d₃-, d₂-, d₁- and d₀-Boldenone sulfate triethylamine salt are also present. The stated mass of the analyte per ampoule represents the combined masses of deuterated (d₃, d₂ and d₁) and d₀-boldenone sulfate triethylamine 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 ₃ ≈ 97% [= d ₃ /(d ₃ + d ₂ + d ₁ + d ₀) × 100] |
| | d ₀ < 0.5% [= d ₀ /(d ₃ + d ₂ + d ₁ + d ₀) × 100] |
| | [from SIM analysis of the free steroid] |

| | | |
|-------|---|---|
| HPLC: | Instrument: | Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler |
| | Column: | X-Bridge C-18, 5 µm (4.6 mm × 150 mm) |
| | Column oven: | 40 °C |
| | Mobile Phase: | Methanol/MilliQ water (44:56 v/v) |
| | Flow rate: | 1.0 mL/min |
| | Detector: | Shimadzu SPD-M20A PDA 2998 operating at Max plot |
| | Relative peak area of the main component: | |
| | Initial analysis: | Mean = 93.0%, s = 0.2% (9 sub samples in duplicate, January 2011) |

Karl Fischer analysis: Moisture content 4.4% mass fraction (December 2011)

Thermogravimetric analysis: Non volatile residue < 0.2% mass fraction. The volatile content (e.g. organic solvents and/or water) could not be determined because of the inherent volatility of the material (December 2011)

Spectroscopic and other characterisation data

| | | |
|----------------------|-----------------------|---|
| LC-MS: | Instrument: | Waters 2695 (HPLC)/Micromass Quatro |
| | Ionisation mode: | Electrospray negative ion |
| | Capillary voltage: | 3.0 kV |
| | Cone voltage: | 20 V |
| | Source temp: | 130 °C |
| | Desolvation gas temp: | 350 °C |
| | Cone gas flow rate: | 23 L/hr |
| | Desolvation gas flow: | 759 L/hr |
| | Dissociated Peak: | 368.0 (M-triethylamine+) <i>m/z</i> |
| IR: | Instrument: | Biorad FTS3000MX FT-IR |
| | Range: | 4000-400 cm ⁻¹ , KBr powder |
| | Peaks: | 3354, 3031, 2976, 2940, 2739, 2677, 2491, 2236, 1658, 1620, 1471, 1446, 1260, 1217, 1015, 980, 821 cm ⁻¹ |
| ¹ H NMR: | Instrument: | Bruker Avance III-400 |
| | Field strength: | 400 MHz |
| | Solvent: | CD ₃ OD (3.31 ppm) |
| | Spectral data: | δ 0.90 (3H, s), 0.93-1.11 (3H, m), 1.29 (3H, s), 1.32 (9H, t, <i>J</i> = 7.3 Hz), 1.13-1.49 (2H, m), 1.63 (1H, dd, <i>J</i> = 7.2, 12.3 Hz), 1.68-1.85 (3H, m), 1.98-2.05 (2H, m), 2.4 (1H, ddd, <i>J</i> = 2.7, 3.8, 13.1 Hz), 2.57 (1H, ddt, <i>J</i> = 1.2, 5.2, 20.2 Hz), 3.22 (6H, q, <i>J</i> = 7.3 Hz), 6.06 (1H, s), 6.21 (1H, dd, <i>J</i> = 1.9, 10.1 Hz), 7.30 (1H, d, <i>J</i> = 10.1 Hz) ppm |
| ¹³ C NMR: | Instrument: | Bruker Avance III-400 |
| | Field strength: | 100 MHz |
| | Solvent: | CD ₃ OD (49.00 ppm) |
| | Spectral data: | δ 9.2, 12.1, 19.1, 23.6, 24.2, 33.8, 34.5, 36.6, 37.6, 44.0, 45.4, 48.0, 50.9, 54.2, 124.0, 127.5, 159.6, 173.5, 188.7 ppm |
| Melting point: | | 97-101 °C |
| Microanalysis: | Found: | C = 61.4%; H = 9.0%; N = 2.9% (November, 2011) |
| | Calculated: | C = 63.8%; H = 9.4%; N = 3.0% (Calculated for C ₂₅ H ₃₈ D ₃ NO ₅ S) |
| | Calculated: | C = 61.0%; H = 9.0%; N = 2.9% (Calculated for C ₂₅ H ₃₈ D ₃ NO ₅ S with 4.4% H ₂ O) |