



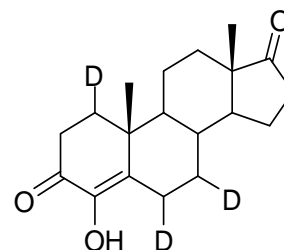
REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA S046: 1,6,7-d₃-4-Hydroxyandrostendione

Report ID: S046.2024.01 (Ampouled 191017)

Chemical Formula: C₁₉H₂₃D₃O₃

Molecular Weight: 305.4 g/mol



Property value

Batch No.	CAS No.	Purity by HPLC-UV
18-S-04	N/A	99.1% (s = 0.02%)

Synonyms: 1,6,7-d₃-Formestane

Expiration of certification: The property values are valid till 29 August 2029, 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 bottles that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials.

Description: White powder prepared by synthesis and certified for identity and purity by NMIA. The main component of this material is d₃-4-hydroxyandrostendione. d₂-, d₁- and d₀-4-Hydroxyandrostendione are also present. The stated purity of the analyte represents the combined masses of deuterated (d₃, d₂ and d₁) and d₀-4-hydroxyandrostendione in the material.

Intended use: This material has not been fully characterized and 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). Each ampoule contains approximately 200 µg of S046.

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: 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.
25 September 2024.

This report supersedes any issued prior to 25 September 2024.

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:

HPLC: Instrument: Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
Column: ACE Super C-18, 5 µm (4.6 mm x 250 mm)
Column oven: 40 °C
Mobile Phase: Methanol/MilliQ water (55:45 v/v)
Flow rate: 1 mL/min
Detector: Shimadzu SPD-M20A PDA operating at 276 nm
Relative peak area of the main component:
Initial analysis: Mean = 98.9%, s = 0.011% (7 sub samples in duplicate, November 2019)
Re-analysis: Mean = 98.9%, s = 0.02% (5 sub samples in duplicate, May 2022)
Re-analysis: Mean = 99.1%, s = 0.02% (5 sub samples in duplicate, August 2024)

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 organic purity value was obtained by HPLC with UV detection.

Supporting evidence is provided by ¹H NMR spectroscopy.

The main component of this material is d₃-4-hydroxyandrostendione. d₂-, d₁- and d₀-4-Hydroxyandrostendione are also present. The stated chemical purity of the analyte represents the combined mass fractions of deuterated (d₃, d₂ and d₁) and d₀-4-hydroxyandrostendione 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: $d_3 \approx 48\% [= d_3 / (d_0 + d_1 + d_2 + d_3) \times 100]$
 $d_0 < 3.0\% [= d_0 / (d_0 + d_1 + d_2 + d_3) \times 100]$

HPLC: Instrument: Shimadzu Binary pump LC-20AB, SIL-20 A HT autosampler
Column: Alltima C-18, 5 µm (4.6 mm x 150 mm)
Column oven: 40 °C
Mobile Phase: Methanol/MilliQ water (60:40 v/v)
Flow rate: 1 mL/min
Detector: Shimadzu SPD-M20A PDA operating at 276 nm
Relative peak area of the main component:
Initial analysis: Mean = 99.7%, s = 0.02% (7 sub samples in duplicate, August 2018)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	DB-5, 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 time of the parent compound is reported along with the major peaks in the mass spectrum. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (11.9 min):	305 (M ⁺ , 75), 263 (19), 262 (15), 164 (16), 160 (16), 148 (100), 140 (32), 114 (75), 93 (27), 68 (20), 55 (27) <i>m/z</i>
TLC:	Conditions:	Kieselgel 60F ₂₅₄ . Hexane/ethyl acetate (65:35) Single spot observed, R _f = 0.3. Visualisation with UV at 254 nm
IR:	Instrument:	Biorad FTS3000MX FT-IR
	Range:	4000-400 cm ⁻¹ , KBr powder
	Peaks:	3380, 2945, 2915, 1735, 1661, 1382, 1257, 1178, 1043, 1011, 709, 651, 616, 585 cm ⁻¹
¹ H NMR:	Instrument:	Bruker Avance III 500
	Field strength:	500 MHz
	Solvent:	MeOH-d ₄ (3.31ppm)
	Spectral data:	δ 0.93 (3H, s), 1.01 (1.4H, m), 1.23 (3H, s), 1.24-1.37 (2H, m), 1.48 (1H, dddd, <i>J</i> = 13.6, 13.6, 13.6, 4.1 Hz), 1.59-1.82 (4.3H, m), 1.96-2.13 (4.3H, m), 2.37-2.49 (2H, m), 2.56 (1H m), 3.08 (0.4H, m) ppm Ethyl acetate estimated at 0.4% mass fraction was observed in the ¹ H NMR.
¹³ C NMR:	Instrument:	Bruker Avance III 500
	Field strength:	500 MHz
	Solvent:	MeOH-d ₄ (49.0 ppm)
	Spectral data:	δ 14.1, 17.4, 21.4, 22.7, 23.5, 31.0, 32.6, 33.2, 35.9, 36.7, 38.9, 52.2, 52.3, 55.8, 141.3, 143.0, 195.3, 223.6 ppm
Melting point:	197-202 °C	