



REFERENCE MATERIAL PRODUCT INFORMATION SHEET

Report ID: D782b.2017.01 (Ampouled 090107)

This batch of ampoules was prepared from the bulk material on 7th January 2009.

Compound Name: **d₃-Nandrolone sulfate (triethylammonium salt)**

Description: Off white solid

Collection Number: D782b

Chemical Formula: C₂₄H₃₈D₃NO₅S

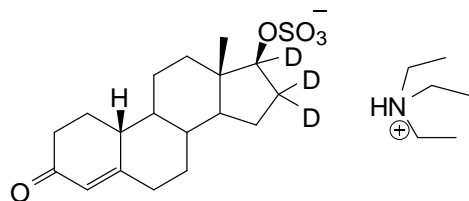
CAS Number: N/A

Structure:

Batch Number: 08-S-04

Molecular Weight: 458.7

Release date: December 2008



Synonyms: (16,16,17 α -d₃)-Nandrolone sulfate, triethylammonium salt
d₃-17 β -sulfooxy-4-estren-3-one triethylammonium salt
d₃-estr-4-en-17 β -ol-3-one triethylammonium salt

The main component of this material is d₃-nandrolone sulfate as a combination of the triethylammonium salt (~78%) and as the protonated sulfate (~22%). d₂-, d₁- and d₀-Nandrolone sulfate are also present. The stated mass of the analyte per ampoule represents the combined masses of deuterated (d₃, d₂ and d₁) and d₀- nandrolone sulfate anion in the material.

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 D782b. Open the ampoule and carefully rinse the interior at least three times with a suitable organic solvent (e.g. methanol). This will transfer approximately 651 μ g of anhydrous nandrolone sulfate anion (d₃, d₂, d₁ and d₀).

This material should be considered for use as an internal standard only.

Isotopic Purity: d₃ \approx 92% $\left[= \frac{d_3}{d_3 + d_2 + d_1 + d_0} \times 100 \right]$

d₀ < 0.5% $\left[= \frac{d_0}{d_3} \times 100 \right]$

(determined by SIM analysis of d₃-nandrolone used to prepare D782b)

HPLC: Instrument: Waters Model 1525 Binary pump, 717 plus autosampler
Column: Alltima C-18, 5 μ m (4.6 mm \times 150 mm)
Mobile Phase: Acetonitrile/ MilliQwater
The aqueous phase was buffered at pH 4.2 using 10mM NH₄OAc
Gradient 0-5 min 75% A, 10-18 min 40% A, 20-26 min 75% A
Flow Rate: 1.0 mL/min
Detector: Waters PDA 2998 operating at 242 nm
Relative peak area response of main component:
Initial analysis: Mean = 95.97%, s = 0.18% (7 ampoules in duplicate, January 2009)
Re-analysis: Mean = 90.1%, s = 0.5% (5 ampoules in duplicate, February 2012)
Re-analysis: Mean = 84%, s = 0.9% (5 ampoules in duplicate, January 2015)
Re-analysis: Mean = 81.4%, s = 1.0% (6 ampoules in duplicate, November 2017)

The following analytical data was obtained on the bulk material subsequently used in the preparation of the ampoules.

The purity value was obtained from traditional analytical techniques. The purity estimate by traditional analytical techniques was obtained by subtraction from 100 % of total impurities by HPLC with UV detection, thermogravimetric analysis, Karl Fischer analysis and ¹H NMR. Supporting evidence is provided by elemental microanalysis.

HPLC:	Instrument:	Waters Model 1525 Binary pump, 717 plus autosampler	
	Column:	Alltima C-18, 5 µm (4.6 mm × 150 mm)	
	Mobile Phase:	Acetonitrile/MilliQ water	
		The aqueous phase was buffered at pH 4.2 using 10mM NH ₄ OAc	
		Gradient 0-5 min 75% A, 10-18 min 40% A, 20-26 min 75% A	
	Flow Rate:	1.0 mL/min	
ESI-MS:	Detector:	Waters PDA 996 at 254 nm	
	Relative peak area response of main component:		
	Initial analysis:	Mean = 96.6%, s = 0.06% (10 sub samples in duplicate, May 2008)	
	Instrument:	Micromass Quattro Micro	
TLC:	Operation:	Negative ion mode, direct infusion at 5 µL/min	
	Ionisation:	ESI spray voltage at 3.0 kV negative ion	
	EM voltage:	650 V	
	Cone voltage:	30 V	
	Peak:	356 (M-Et ₃ NH ⁺) <i>m/z</i>	
IR:	Conditions:	Kieselgel 60F ₂₅₄ . Chloroform/methanol (17/3)	
		Single spot observed, R _f = 0.32. Visualisation with UV at 254 nm	
¹ H NMR:	Instrument:	Biorad FTS300MX FT-IR	
	Range:	4000-400 cm ⁻¹ , KBr pellet	
	Peaks:	3034, 2909, 2518, 2361, 1680, 1622, 1472, 1260, 1196, 602 cm ⁻¹	
¹³ C NMR:	Instrument:	DMX 600	
	Field strength:	600 MHz	Solvent: CDCl ₃
	Spectral data:	δ 0.85 (3H, s), 0.8-0.9 (1H, m), 1.0-1.1 (2H, m), 1.2-1.4 (4H, m) 1.41 ("6.7" H, t, <i>J</i> = 7.3 Hz), 1.51 (1H, m), 1.62 (1H, dd, <i>J</i> = 7.3, 12.3 Hz), 1.80 (2H, m), 1.97 (1H, m), 2.07 (1H, m), 2.2-2.3 (3H, m), 2.40 (1H, m), 2.48 (1H, dd, <i>J</i> = 1.5, 7.9 Hz), 3.05 ("4.7" H, t, <i>J</i> = 7.3 Hz) 5.83 (1H, s) ppm	
¹³ C NMR:	Instrument:	DMX 600	
	Field strength:	150 MHz	Solvent: d ₄ -MeOH (48.0 ppm)
	Spectral data:	δ 10.6, 11.1, 22.9, 26.1, 26.7, 30.9, 35.5, 36.3, 36.7, 40.4, 42.6, 42.7, 42.9, 49.5, 50.0, 123.8, 169.8, 201.9 ppm	
Thermogravimetric analysis:		Initial volatile content < 0.1% and non volatile residue < 0.2% mass fraction (July 2008)	
Karl Fischer analysis:		Moisture content 0.3% mass fraction (February 2008)	

Expiration of certification

The property values are valid till 8th November 2020, i.e. three 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.

The long-term stability of the compound in solution has not been examined.

In the absence of 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 10 years.

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.

Intended use

For *in vitro* laboratory analysis only.

Caution

Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice

Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 21 November, 2017.

Characterisation data and property values specified in this report supersede those in all reports issued prior to 21st November 2017.