Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

CERTIFIED REFERENCE MATERIAL
CERTIFICATE OF ANALYSIS

NMIA MX003: 19-Norandrosterone in 1,2-dimethoxyethane solvent

Certified values

Steroid [#]	CAS No.	Mass fraction (ng/g)	Mass concentration (ng/mL)*	Coverage Factor (k)
19-norandrosterone	**1225-01-0**	**221.4 ± 6.9**	**192.0 ± 6.0**	**2.0**

The measurand is defined as the free steroid form of 19-norandrosterone in the solution in ng/g or ng/mL.

**This mass concentration applies to 20 °C. The conversion from mass fraction units of ng/g to mass concentration units of ng/mL at 20 °C was carried out by measuring the density of the material. The density was measured as 866.98 ± 0.05 kg/m3 at 20 °C (± 0.02 °C).*

Batch: 2004.01

Expiry: 30 December 2023

Description: This reference material is 1 mL of a solution of 19-norandrosterone (CAS Registry Number 1225-01-0) in 1,2-dimethoxyethane solvent (CAS Registry number 110-71-4) contained in a sealed glass ampoule.

Intended use: The reference material is intended to be used as a calibration standard for analytical methods for the measurement of 19-norandrosterone, a metabolite of 19-nortestosterone (nandrolone), in doping, clinical or forensic analysis.

Instructions for use: Break the top off the ampoule at the etch mark and use immediately. The solvent is volatile and significant evaporation may occur that will change the concentration value and this should be avoided.
The smallest subsample used for homogeneity assessment was 0.35 g; the use of a smaller quantity may expand the stated uncertainties for the certified values.

Storage: Store at -20 °C out of direct light in the sealed ampoule as issued.

Metrological traceability: The certified values are traceable to the SI unit for mass (kg) through the Australian national standards for mass. The primary ratio method of isotope dilution mass spectrometry (IDMS) was used and the quantities used in the calculation of the measurement values are traceable to the SI. The pure steroid standards used in the calibration of the IDMS method were traceable through certification. Balances used in the preparation of samples were all appropriately calibrated.
The certified mass concentrations are traceable to the SI units for mass (kg) and length (m) through the relevant Australian national standards. The mass fraction values obtained by IDMS have been converted to mass concentration using a traceable density measurement carried out at NMIA's mass laboratory.

Stability: The stability of the material under the recommended storage conditions has been verified via a protocol compliant with ISO Guide 35:2017[1] and will continue to be monitored. Long term stability was assessed at -20 °C. No instability was observed between -20 °C and 40 °C.

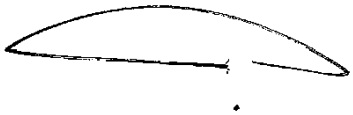
Homogeneity: The homogeneity of the material was assessed from 33 ampoules selected in a stratified sampling plan to test for homogeneity across the entire batch. Both within- and between-ampoule variance was estimated. Homogeneity testing was carried out on 0.35 g aliquots of the material. Both within- and between-bottle variance was estimated. The uncertainties in the certified values incorporate these results.

Safety: CRM NMIA MX003 is intended for in-vitro diagnostic analysis only.

Production: A stock solution was prepared on 1 September 2004 by dissolving 10 mg of 19-norandrosterone (NMIA D555) in 10 g of 1,2-dimethoxyethane (glyme). A portion (0.29 g) of this solution was diluted to 1305 g with glyme then dispensed into ampoules and flame sealed. The certified value agreed with the calculated gravimetric mass fraction of the solution.

Analytical method: The certified mass fraction was measured by isotope dilution mass spectrometry involving the addition of an isotopically-labelled analogue of 19-norandrosterone prior to any sample preparation. The mass fraction determined analytically by GC-HRMS was converted to a mass concentration from the measured density of the material.

Measurement uncertainty: Standard uncertainties were estimated and combined as described in the JCGM Guide to the Expression of Uncertainty in Measurement.[2] The individual components contributing to the measurement uncertainty estimates were the uncertainties associated with the calibration standards, gravimetric weighings, method precision, heterogeneity between ampoules, storage stability of the material at -20 °C and bias in the reference analytical procedure. The combined standard uncertainties were expanded to a level of confidence of 95% using a coverage factor calculated from the effective degrees of freedom obtained from the Welch-Satterthwaite equation.



Mark Lewin
Technical Manager, Chemical Reference Values
8 March 2022

Accreditation No.198

The property values specified in this report supersede any issued prior to 8 March 2022

References:

1. Reference Materials – Guidance for characterisation and assessment of homogeneity and stability. ISO Guide 35:2017
2. Joint Committee for Guides in Metrology; Evaluation of measurement data — Guide to the Expression of Uncertainty in Measurement; JCGM 100:2008.

CIPM MRA Notice: This certificate is consistent with the capabilities that are included in Appendix C of the CIPM MRA drawn up by the CIPM. Under the CIPM MRA, all participating institutes recognize the validity of each other's calibration and measurement certificates for the quantities, ranges and measurement uncertainties specified in Appendix C. The "CIPM MRA Logo" and this statement attest only to the measurement(s) applied for determining the certified values on the certificate (for details see http://www.bipm.org).

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

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