Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

CIPM MRA

NATA
ACCREDITED FOR TECHNICAL COMPETENCE

CERTIFIED REFERENCE MATERIAL
CERTIFICATE OF ANALYSIS

NMIA MX021: Formestane certified for Carbon Isotope Delta Value

Certified value

Steroid	CAS No.	$\delta^{13}C_{VPDB}$ / ‰	Coverage Factor (k)	V_{eff}
Formestane	**566-48-3**	**–30.71 ± 0.48**	**2.0**	**34**

The measurand is the carbon isotope ratio delta value of the stated steroid relative to that embodied in the primary isotopic reference material VPDB. The uncertainties are expanded to provide a level of confidence of 95%.

Expiry: 30 August 2026 **Batch No.:** 2019.08

Description: 2 mL sealed ampoule containing approximately 200 µg of pure Formestane.

Intended use: Validation and calibration of Gas Chromatography Combustion Isotope Ratio Mass Spectrometry (GC-C-IRMS) for steroid carbon isotope ratio measurements in anti-doping analysis.[1] In-vitro use only.

Instructions for use: Each ampoule must be reconstituted in a minimum of 1 mL of an appropriate solvent such as 2-propanol and allowed to equilibrate for 3 hours before use.

Storage: Store in a refrigerator at 4°C out of direct light.

Metrological traceability: The certified carbon isotope delta value is traceable to the stable carbon isotope ratios of the primary isotopic reference material NBS19 via the secondary isotopic reference materials USGS-40 (L-glutamic acid) and IAEA-CH-7 (polyethylene).[2]

Stability: The stability of the material under the recommended storage conditions has been verified by GC-C-IRMS and will continue to be monitored. The material was stable in an accelerated stability trial of two weeks at 40°C.

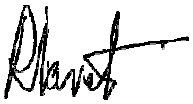
Homogeneity: Fifteen ampoules were selected using a stratified sampling plan and each ampoule was analysed twice by GC-C-IRMS. No significant inhomogeneity was detected.

Production: The stock solution for the mixture was prepared by dissolving nominally 0.25 g of Formestane in 250 mL of a mixture of 2-propanol and methanol prior to dispensing 0.2 mL aliquots into ampoules. The ampoules were vacuum dried for two days then flame sealed under argon and stored at 4°C.

Analytical method: EA-IRMS was employed to assign $\delta^{13}C_{VPDB}$ value to the pure steroid material used for preparation of MX021. Calibration was performed using a two-point normalization approach[3] with USGS-40 and IAEA-CH-7 providing metrological traceability to the VPDB scale. The uncertainty contribution from scale calibration and ^{17}O correction by instrument software was estimated in each batch of analysis by using two CRMs (NBS 22 and IAEA-600) as quality control samples.

Measurement uncertainty: Standard uncertainties were estimated and combined as described in the JCGM Guide to the Expression of Uncertainty in Measurement.[4] The combined standard uncertainties were expanded with coverage factors calculated from degrees of freedom obtained from Welch-Satterthwaite equation. Based on the purity analysis of the starting materials the

potential for bias was estimated and included in the expanded measurement uncertainty. The major contributions to the combined measurement uncertainty were potential biases, between-unit homogeneity, measurement reproducibility, potential instability due to transport conditions and uncertainty in the $\delta^{13}C_{VPDB}$ values of the calibration materials.



Raluca Iavetz
Manager Chemical Reference Values
30 August 2021
Report ID: MX021.2021.01

Accreditation No. 198

References:

1. WADA. *Technical Document - TD2021IRMS: Detection of Synthetic Forms of Endogenous Anabolic Androgenic Steroids, Version 1.0*
2. IUPAC *Technical Report: Assessment of international reference materials for isotope-ratio analysis,* Pure Appl Chem, 2014, 86, 425-467
3. Debajyoti P, Skrzypek G and Istvan F, Normalisation of measured stable isotopic compositions to isotope reference scales – a review, Rapid Comm in Mass Spectrometry, 2007; 21:3006-3014.
4. JCGM, *Evaluation of measurement data — Guide to the expression of uncertainty in measurement*. JCGM100:2008

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*).

This Certificate for NMIA MX021 supersedes any issued prior to 30 August 2021.

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