

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 6212-a

No. +++

 3β , 4α -Dihydroxy- 5α -Androstan-17-One Standard Solution

This certified reference material (CRM), 3β , 4α -dihydroxy- 5α -androstan-17-one solution (dissolved in methanol), is produced in accordance with the NMIJ's management system and is in compliance with ISO 17034 and ISO/IEC 17025. This CRM is intended for the calibration of instruments and validation of analytical methods.

Certified Values

The certified values of this CRM, which are mass fraction and mass concentration of 3β , 4α -dihydroxy- 5α -androstan-17-one, are given in the tables below. The uncertainty of the certified values are the expanded uncertainties obtained by multiplying the combined standard uncertainties by a coverage factor (k) of 2, and it is the half-width of an interval estimated to have a level of confidence of approximately 95 %.

Substance	Certified value Mass fraction ($\mu\text{g/g}$)	Expanded uncertainty Mass fraction ($\mu\text{g/g}$)
3β , 4α -Dihydroxy- 5α -androstan-17-one	135.2	3.8

Substance	Certified value Mass concentration ($\mu\text{g/mL}$)	Expanded uncertainty Mass concentration ($\mu\text{g/mL}$)
3β , 4α -Dihydroxy- 5α -androstan-17-one	107.0	3.0

The certified value for mass concentration is the value at 20 °C.

Analysis

The certified value (mass fraction) of this CRM was an arithmetic mean of the following values:

- (1) Preparation value based on gravimetric blending: The mass fraction was calculated from the purity of 3β , 4α -dihydroxy- 5α -androstan-17-one raw material and the dilution rate adopted in the preparation of this CRM. The purity of 3β , 4α -dihydroxy- 5α -androstan-17-one was an arithmetic mean of values determined by quantitative Nuclear Magnetic Resonance spectroscopy (qNMR) and a method combining qNMR and high-performance liquid chromatography (qNMR/HPLC).
- (2) Measured value: The value was an arithmetic mean of mass fractions of the compound in the CRM determined directly by the qNMR and the qNMR/HPLC.

The certified value (mass concentration) was obtained by multiplying mass fraction of 3β , 4α -dihydroxy- 5α -androstan-17-one with the density of this CRM measured at 20 °C by a vibration-type density meter.

The uncertainties derived from purity, preparation, measurement of solution, differences between the measuring methods (qNMR and qNMR/HPLC), homogeneity, and stability were combined to obtain the uncertainty of the certified value (mass fraction). In addition, the uncertainty derived from variation of its density at temperatures of 15 °C to 25 °C was also combined to obtain the uncertainty of the certified value (mass concentration).

Metrological Traceability

The certified value (mass fraction) was obtained by the preparation value and the measured value. The purity of the raw material used for calculating the preparation value and the measured value were obtained by the qNMR and the qNMR/HPLC, both of

which were calibrated by using 1,4-bis(trimethylsilyl)benzene-*d*₄ as an internal standard calibrated with 3,5-bis(trifluoromethyl)benzoic acid (NMIJ CRM 4601-a or NMIJ CRM 4601-b). Balances calibrated in the JCSS (Japan Calibration Service System) were used for gravimetric blending. The density meter was calibrated by a certified density standard solution (JCSS). Therefore, the certified values are traceable to the International System of Units (the SI).

Expiration of Certification

This certificate is valid for one year from the date of shipment, provided that this CRM remains unopened and is stored in accordance with the instructions given in this certificate.

Description of the Material

This CRM is a methanol solution containing 3 β , 4 α -dihydroxy-5 α -androstan-17-one and in the form of a colorless clear liquid at room temperature. Approximately 1 mL of this CRM is bottled in a brown glass ampoule.

Instructions for Storage

This CRM should be stored at temperatures of -30 °C to -15 °C and protected from light.

Instructions for Use

This CRM should be used at temperatures of 15 °C to 25 °C when it is used for mass concentration. The ampoule should be allowed to warm to room temperature before opening. Prior to use, the ampoule should be shaken thoroughly but gently at room temperature. This CRM should be used up promptly once an ampoule is opened while care should be taken for volatilization of methanol. This CRM should not be used for tests of living bodies or any other purposes than laboratory use.

Precautions for Handling

This CRM should be handled in a well-ventilated place while it is protected from fires. A mask, gloves, and other personal protective equipment should be used when this CRM is handled. Refer to the safety data sheet (SDS) on this CRM before use. Care should be taken for injury when the glass ampoule is opened. This CRM should be disposed of in accordance with applicable local rules and regulations.

Preparation

The raw material, 3 β , 4 α -dihydroxy-5 α -androstan-17-one synthesized by FUJIFILM Wako Pure Chemical Corporation, was dissolved in methanol and divided into brown glass ampoules with argon gas at NMIJ.

NMIJ Analysts

The technical manager for this CRM is NUMATA M., the production manager is KUROE M., and the analysts are KUROE M., YAMAZAKI T., SAITO N., SHIMIZU Y., IHARA T., NAKAMURA S., ISHIKAWA K., BAO X., and YAMANAKA N.

Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the registered customers. Customer registration on the NMIJ Website (given below) will facilitate notification. Technical reports regarding this CRM can be obtained from the contact details given below.

Reproduction of Certificate

In reproducing this certificate, it should be clearly indicated that the document is a copy.

Note

This CRM was developed in collaboration with Research Laboratory for Calibration Standards in Doping Analysis, National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology.

April 1, 2020

ISHIMURA Kazuhiko
President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:
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Revision history

September 17, 2020: The uncertainties were changed by the results of stability monitoring.

September 28, 2023: The description in “Expiration of Certification” was changed to “one year from the date of shipment.”
“Technical Information”, was removed.