National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate NMIJ CRM 6015-a No. +++



∟-Valine

This certified reference material (CRM) is produced in accordance with the NMIJ's management system and is in compliance with ISO 17034 and ISO/IEC 17025. It is primarily intended for use in calibrating the analytical instruments or reagents in amino acid analysis. It is also intended for controlling the precision of analysis, and for the validating analytical methods and instruments.

Certified Values

The certified value for the purity (in mass fraction) of L-valine is given in the table below. The uncertainty of the certified value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, which gives a level of confidence of approximately 95 %.

| | CAS No. | Certified value, Mass fraction (kg/kg) | Expanded uncertainty, Mass fraction (kg/kg) |
|---|---------|---|--|
| L-Valine ((2 <i>S</i>)-2-amino-3-methylbutanoic acid) | 72-18-4 | 0.998 | 0.002 |

The certified value for the purity (in mass fraction) of valine without enantiomeric separation is given in the table below, where the mass fraction of D-valine is negligible. The uncertainty of the certified value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, which gives a level of confidence of approximately 95 %.

| | Certified value, Mass fraction (kg/kg) | Expanded uncertainty, Mass fraction (kg/kg) |
|---|---|--|
| Valine (without enantiomeric separation) | 0.998 | 0.002 |

Analysis

The certified value is based on the results of acidimetric titration, nitrogen determination by the Kjeldahl method and impurity determination by high performance liquid chromatography (HPLC). Impurities of amino acids were quantified by liquid chromatography mass spectrometry (LC/MS) and by LC with fluorescence detection after derivatization using *o*-phthalaldehyde (OPA). D-valine was quantified by LC/MS using a chiral resolution column.

Metrological Traceability

The certified value was determined by titrimetry as the primary method of measurement with NMIJ CRM 3001-a (potassium hydrogen phthalate) and NIST SRM 351a (sodium carbonate) as primary standards and by impurity determination using HPLC calibrated with purity-evaluated amino acids. It is traceable to the International System of Units (SI).

Mutual Recognition Arrangement under Meter Convention

The certified value and expanded uncertainty of this CRM without enantiomeric separation (mass fraction) is consistent with

Date of Shipment: Xxxxx XX, 20XX

the calibration and measurement capabilities (CMCs) that are included in Appendix C of the Mutual Recognition Arrangement (MRA) drawn up by the International Committee for Weights and Measures (CIPM). Under the 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 (as for Appendix C of MRA, see http://kcdb.bipm.org/AppendixC/default.asp).

Expiration of Certification

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

Sample Form

This CRM is in the form of a white powder. This CRM of 0.5 g in net volume is kept in an amber glass vial and the vial is sealed in an aluminum-laminated plastic bag.

Homogeneity

The homogeneity of this CRM was measured by acidimetric titration, analyzing 10 vials randomly selected from 400 vials. The homogeneity is reflected in the uncertainty of the certified value.

Instructions for Storage

This CRM should be stored at a temperature between 15 °C and 25 °C in a clean desiccator and shielded from light.

Instruction for Use

Considering the homogeneity, a minimum sample mass of 100 mg should be used. This CRM should be used promptly once the vial is opened. The CRM is for laboratory use only and not for in vivo use.

Precautions for Handling

Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

Preparation of the material was performed by Wako Pure Chemical Industries, Ltd. Highly purified L-valine provided by AJINOMOTO Co., Inc. was bottled into vials under argon atmosphere and each vial was sealed in an aluminum-laminated bag.

Technical Information

Impurities which were determined by HPLC at the time of certification, were alanine, isoleucine, leucine and lysine and their mass fractions were 0.41 g/kg, 0.42 g/kg, 0.22 g/kg, and 0.11 g/kg, respectively. At the time of certification, moisture was determined by Karl Fischer titration to be 0.08 g/kg and the mass fraction of D-valine determined by LC/MS using a chiral resolution column was less than 2 mg/kg.

NMIJ Analysts

The technical manager for this CRM is TAKATSU A. and production manager is KATO M. The analysts are KATO M., KATO H., EYAMA S., GOTO M. and YOSHIOKA M.

Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the registered customer. 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.

6015a00-091208-200401

April 1, 2020

ISHIMURA Kazuhiko President National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact: National Institute of Advanced Industrial Science and Technology, National Metrology Institute of Japan, Center for Quality Management of Metrology, Reference Materials Office, 1-1-1 Umezono, Tsukuba, Ibaraki 305-8563, Japan Phone: +81-29-861-4059; Fax: +81-29-861-4009, https://unit.aist.go.jp/nmij/english/refmate/

Revision history

April 1, 2015: "Metrology Management Center" was renamed to "Center for Quality Management of Metrology."Oct. 17, 2018: The description on "Mutual Recognition Arrangement under Meter Convention" was added. The description in "Expiration of Certification" was changed to "one year from the date of shipment."

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