

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 3682-a
No. +++

Iron Isotopic Standard Solution

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. This CRM is intended as a standard for isotope ratio measurement in mass spectrometer.

Certified Values

The certified values of isotopic ratio for this CRM are given in the table below.

	Certified value Isotopic ratio (mol/mol)	Expanded uncertainty Isotopic ratio (mol/mol)
$^{56}\text{Fe}/^{54}\text{Fe}$	15.7233	0.0065
$^{57}\text{Fe}/^{54}\text{Fe}$	0.36346	0.00023
$^{58}\text{Fe}/^{54}\text{Fe}$	0.048225	0.000053

The certified values of isotopic abundance and molar mass calculated from the isotopic ratios are given in the tables below. The data for relative mass of iron required to calculate the molar mass was acquired from "Atomic Weights of the Elements: Review 2000" (J.R.de. Laeter et. al., *Pure Appl.Chem.* **75** (2003) 683.).

	Certified value Isotopic abundance (mol/mol)	Expanded uncertainty Isotopic abundance (mol/mol)
^{54}Fe	0.058360	0.000022
^{56}Fe	0.917614	0.000034
^{57}Fe	0.021212	0.000015
^{58}Fe	0.002814	0.000003

	Certified value Molar mass (g/mol)	Expanded uncertainty Molar mass (g/mol)
Fe	55.845340	0.000038

The uncertainties of the certified values are 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 %.

Analysis

The certified values were determined by isotopic ratio measurements of $^{56}\text{Fe}/^{54}\text{Fe}$, $^{57}\text{Fe}/^{54}\text{Fe}$, and $^{58}\text{Fe}/^{54}\text{Fe}$ by using multiple-collector inductively-coupled plasma mass spectrometer. Mass discrimination effects in the mass spectrometer were corrected by a bracketing method, using a [$^{54}\text{Fe}+^{57}\text{Fe}$] mixed-enriched isotope solution. In the bracketing method, correction factors for the measured $^{56}\text{Fe}/^{54}\text{Fe}$ and $^{58}\text{Fe}/^{54}\text{Fe}$ were estimated from the true value of $^{57}\text{Fe}/^{54}\text{Fe}$ in accordance with Exponential Law. The amount-of-substance ratio of $^{57}\text{Fe}/^{54}\text{Fe}$ in the [$^{54}\text{Fe}+^{57}\text{Fe}$] mixed-enriched isotope solution was accurately determined as follows:

- (1) The ratio of total iron amount-of-substance content in a ^{54}Fe enriched isotope solution to that in a ^{56}Fe enriched isotope solution was determined by EDTA titrimetry.

- (2) The ratio obtained in (1) was corrected by metal impurities, except iron, affecting the EDTA titrimetry in each enriched isotope solution, which were determined by inductively coupled plasma sector-field mass spectrometry (ICP-SFMS).
- (3) Isotopic abundance of minor iron isotopes in each enriched isotope solution was determined by a standard addition method (ICP-SFMS) by using ^{56}Fe enriched spike solution.
- (4) A mixture of the two enriched isotope solutions was made by a gravimetric preparation method, and then the amount-of-substance ratio of $^{57}\text{Fe}/^{54}\text{Fe}$ in the mixed-enriched isotope solution was corrected by the results of (3).

Metrological Traceability

The [$^{54}\text{Fe}+^{57}\text{Fe}$] mixed-enriched isotope solution used for correction of the mass discrimination effects was gravimetrically prepared from a ^{54}Fe enriched isotope solution and a ^{57}Fe enriched isotope solution. The ratio of iron amount-of-substance content of the two enriched isotope solutions was determined by the EDTA titrimetry which is one of the primary methods of measurements. The ratio was corrected by the amount of substance of metal impurities, except iron, affecting the EDTA titrimetry. The metal impurities were determined by a calibration method using ICP-SFMS. The commercially-available standard solutions used for the calibrations were traceable to NIST SRMs. In addition, isotopic abundance of minor iron isotopes in each enriched isotope solution was determined by a standard addition method (ICP-SFMS) using ^{56}Fe enriched spike solution, whose amount of substance of iron was determined by the EDTA titrimetry using NMIJ CRM 3611-a iron standard solution. The certified values, therefore, are traceable to the International System of Units (SI).

Indicative value

The mass fraction of iron determined by the EDTA titrimetry using NMIJ CRM 3611-a iron standard solution is given in the table below as an indicative value. Its uncertainty 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 %.

	Indicative value Mass fraction (mg/kg)	Expanded uncertainty Mass fraction (mg/kg)
Fe	1001.0	3.0

Expiration of Certification

This certificate is valid until March 31, 2024, provided that the CRM remains unopened and is stored in accordance with the instructions given in this certificate.

Description of the material

This CRM of ca. 100 mL in net volume is kept in a high-density polyethylene bottle equipped with an inner lid. The bottle is sealed in an aluminum-laminated plastic bag.

Homogeneity

The homogeneity of this CRM was determined by analyzing 10 bottles selected from among 245 bottles by the stratified random sampling method. If the solution is mixed enough prior to use, the homogeneity within the bottle is good enough for normal purpose. The homogeneity has been incorporated in the uncertainties of the certified values and indicative value.

Instructions for Storage

The solution of this CRM should be kept in the high-density polyethylene bottle sealed in aluminum-laminated plastic bag. This CRM should be stored in a clean place at temperatures of 10 °C or less while keeping this CRM unfrozen.

Instructions for Use

The bottle of this CRM should be allowed to warm to room temperature before opening. Prior to use, the bottle should be shaken thoroughly but gently at room temperature.

Precautions for Handling

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

Preparation

This CRM was prepared from high-purity iron reference material JSS 001-8 (The Japan Iron and Steel Federation). The high-purity iron was dissolved in nitric acid and diluted to 27 kg of approximately 1.4 % (mass fraction) nitric acid. The solution was divided into 100-mL bottles; each bottle contains approximately 100 mL of the solution.

Technical Information

When $^{56}\text{Fe}/^{54}\text{Fe}$, $^{57}\text{Fe}/^{54}\text{Fe}$, and $^{58}\text{Fe}/^{54}\text{Fe}$ of this CRM were measured by the bracketing method using IRMM-014, delta values to the certified values of IRMM-014 were -2.189, -3.255 and -4.516, respectively. The density of the solution used for air-buoyancy correction was 1.006 g/cm^3 (25 °C).

NMIJ Analysts

The technical manager for this CRM is MIURA T., the production manager is NONOSE N., and the analysts are NONOSE N., SUZUKI T. and ISHIZAWA Y.

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 produced with the support of Joint Research Grant for Environmental Isotope Study of the Research Institute for Humanity and Nature (RIHN).

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

February 9, 2022: The description in “Expiration of Certification” was changed to “until March 31, 2024”.