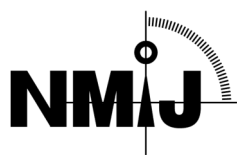


National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 8004-a
No. +++

Fine Silicon Nitride Powder for Fine Ceramics (Direct Nitridation) II

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 for use in controlling the precision of analysis or confirming the validity of analytical methods and instruments during the quantitative determination of main constituents and trace elements in silicon nitride.

Certified Values

The certified values of 10 elements in this CRM are given in the tables below. The drying method is described in this certificate. 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 %.

Main constituents

Element	Certified value, Mass fraction (%)	Expanded uncertainty, Mass fraction (%)	Analytical method
Total Si	59.23	0.19	condensation / gravimetric method with correction by inductively coupled plasma atomic emission spectrometry
Total N	38.49	0.22	pressurized acid digestion - Kjeldahl distillation / acidimetric titration method

Trace metals

Element	Certified value, Mass fraction (mg/kg)	Expanded uncertainty, Mass fraction (mg/kg)	Analytical method*
Al	740	18.0	1), 3), 4)
Ca	72.7	4.6	1), 2), 3), 4)
Fe	196.9	6.0	1), 2), 3), 4)
Mg	10.29	0.40	1), 2), 3), 4)
Mn	2.99	0.14	1), 3), 4)
Ni	2.49	0.17	1), 2), 3), 4)
Ti	8.52	0.26	1), 2), 3), 4)
Zr	2.15	0.10	1), 2), 3), 4)

* Analytical method

1) pressurized acid digestion / inductively coupled plasma mass spectrometry, 2) pressurized acid digestion / isotope dilution – inductively coupled plasma mass spectrometry, 3) pressurized acid digestion / inductively coupled plasma optical emission spectrometry, 4) alkaline fusion / inductively coupled plasma optical emission spectrometry.

Analysis

Each certified value was determined as a weighted mean of more than one analytical result. The main constituents were determined by a primary method of measurement. The metal impurities were determined by combination of a primary method of measurement and other methods proved to be valid or combination of more than two methods proved to be valid. The

analytical methods of metal impurities include multiple digestion methods and multiple instrumental measurements. The expanded uncertainty includes the combined uncertainty due to analytical results obtained by multiple analytical methods, homogeneity and stability.

Metrological Traceability

The certified value of total Si was determined by gravimetric analysis as a primary method of measurement. The certified value of total N was determined by acidimetric titration method with NMIJ CRM 3001-a (potassium hydrogen phthalate) as a primary reference material. Each certified value of metal impurities was determined by more than one methods including isotope dilution – mass spectrometry as a primary method of measurement with NMIJ primary standard solutions, JCSS standard solutions, or NIST SRM standard solutions. The certified values are traceable to the International System of Units (SI).

Indicative Values

The indicative values of 7 kinds of elements in this CRM are given in the table below. In the analysis of these elements, only single digestion method or single instrumental measurement was employed. The expanded uncertainty (a coverage factor, $k = 2$) includes the uncertainty due to the analytical result, homogeneity and stability.

Trace metals

Element	Indicative value, Mass fraction (mg/kg)	Expanded uncertainty, Mass fraction (mg/kg)	Analytical method*
Ba	6.26	0.58	3), 4)
Co	5.99	0.30	1), 3)
Cr	9.70	0.36	1), 2), 3)
Cu	0.71	0.28	1), 2)
Mo	2.59	0.32	1), 2), 3)
Sr	0.846	0.066	3), 4)
Y	38.8	1.6	1), 3)

* Analytical method

1) pressurized acid digestion / inductively coupled plasma mass spectrometry, 2) pressurized acid digestion / isotope dilution – inductively coupled plasma mass spectrometry, 3) pressurized acid digestion / inductively coupled plasma optical emission spectrometry, 4) alkaline fusion / inductively coupled plasma optical emission spectrometry.

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 fine powder and it of ca. 25 g in net volume is kept in a glass bottle.

Homogeneity

The homogeneities of the trace metal elements in this CRM were determined by analyzing 16 bottles chosen from approximately 400 bottles. The elements were determined by pressurized acid digestion / inductively coupled plasma optical emission spectrometry. The homogeneity of main constituents and non-metal elements was evaluated from the repeatability of quantitative analysis. The homogeneity of each element is reflected in the uncertainty of the certified values and the indicative values.

Instructions for Storage

This CRM should be tightly sealed, stored at a temperature between 15 °C and 35 °C away from a high humidity and shielded from light.

Instructions for Use

- (1) Prior to use, the sample should be mixed by slow rolling; however, it should not be mixed vigorously in order to minimize the risk of shaving the container wall.
- (2) From the homogeneity, the recommended sample mass for one analysis is 300 mg or more for total Si, 150 mg or more for total N, 15 mg or more for O, and 500 mg or more for the others.
- (3) The certified values and information ones are shown as those for the dried material. The recommended drying method is as follows:
 - ① The material should be dried for two hours at 110 °C.
 - ② The material should be stood at room temperature in magnesium perchlorate desiccator and weighed as soon as possible after cooling.

Precautions for Handling

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

Preparation

A typical commercial fine silicon nitride powder for fine ceramics was purchased. The material of 25 g each was packed in approximately 400 glass bottles.

Technical Information

In this CRM, the determined value of fluorine obtained by pyrohydrolysis / ion-chromatography was 3 mg/kg as mass fraction at the certification on January 2008, and that of oxygen obtained by inert gas fusion / IR spectrometry was 1.4 % as mass fraction at the stability monitoring on February 2016.

NMIJ Analysts

The technical managers are UWAMINO Y. and HIOKI A., the production manager is UWAMINO Y., and the analysts are UWAMINO Y., HIOKI A., TSUGE A., MORIKAWA H. and NONOSE N.

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.

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."

December 21, 2015: The indicative value of yttrium was revised.

July 19, 2016: The values of oxygen and fluorine impurities were changed from "Indicative value" to "Information."
Identified figures of the certified values were changed as a result of rounding to two significant figures of their uncertainties.

The description in "Expiration of Certification" was changed to "one year after the date of shipment."

Sample