

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 8001-a  
No. +++Fine Silicon Carbide Powder for Fine Ceramics ( $\alpha$ -phase)

This certified reference material (CRM) is fine silicon carbide powder for fine ceramics ( $\alpha$ -phase), is produced in accordance with the NMIJ's management system and in compliance with ISO 17034 and ISO/IEC 17025. This CRM is intended for use in controlling the precision of analysis and validating analytical methods and instruments during the analysis of main constituents and trace elements in silicon carbide.

**Certified Values**

The certified values of 6 elements in this CRM are given in the tables 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 %.

**Main constituents**

Element	Certified value, mass fraction (%)	Expanded uncertainty, mass fraction (%)	Analytical method
Total Si	68.31	0.58	condensation / gravimetric method with correction by inductively coupled plasma atomic emission spectrometry
Total C	29.80	0.15	dry combustion / gravimetric method

**Trace metals**

Element	Certified value, mass fraction (mg/kg)	Expanded uncertainty, mass fraction (mg/kg)	Analytical method (*vide infra)
Al	83.2	7.2	2), 3), 4)
Fe	46.7	7.8	1), 2), 3), 4)
Ti	6.37	0.68	1), 2), 3), 4)
Y	0.31	0.066	2), 3), 4)

\* analytical method

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

**Analysis**

Each certified value was determined as a weighted mean of more than one analytical result in NMIJ and the Research Institute of Instrumentation Frontier (RIIF). The expanded uncertainty includes the combined uncertainty due to analytical results obtained by multiple analytical methods and homogeneity.

**Metrological Traceability**

The analytical method(s) were as follows and each certified value is traceable to the International System of Units (SI):

- (1) a primary method of measurement (for main constituents).

(2) combination of a primary method of measurement and another method proved to be valid.

(3) combination of more than two methods proved to be valid.

### Indicative Values

The indicative values of eleven kinds of elements in this CRM are given in the two tables below. Each value following symbol of  $\pm$  is the half-width of the expanded uncertainty interval, estimated from the combined standard uncertainty and coverage factor ( $k$ ) of 2, which gives a level of confidence of approximately 95 %.

#### Trace metals

Element	Indicative value, mass fraction (mg/kg)	Analytical method (*vide infra )
Cr	$1.98 \pm 0.52$	1), 2), 3)
Cu	$0.47 \pm 0.17$	1), 2), 3), 4)
La	$0.71 \pm 0.32$	2), 3)
Mn	$0.53 \pm 0.092$	2), 3)
Ni	$1.52 \pm 0.22$	1), 2)

\* analytical method

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

#### Trace non-metals

Element	Indicative value, mass fraction (%)	Analytical method
Total free carbon	$0.53 \pm 0.14$	combustion / IR spectrometry
Free carbon from combustion at 550 °C	$0.49 \pm 0.08$	combustion / IR spectrometry
Free carbon from combustion at 850 °C	less than or equal to 0.11	combustion / IR spectrometry
Element	Indicative value, mass fraction (mg/kg)	Analytical method
F	$700 \pm 160$	thermal hydrolysis / ion-chromatography
Cl	$160 \pm 42$	thermal hydrolysis / ion-chromatography
S	$32 \pm 12$	thermal hydrolysis / ion-chromatography

### Expiration of Certification

The certification of this CRM is valid until March 31, 2024, provided that the material 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. 50 g in net volume is kept in a glass bottle.

### Homogeneity

The homogeneities of trace metal elements in the CRM were evaluated together with the determinations themselves by analysing 6 bottles selected from 600 bottles at approximately same intervals in the order of bottling. The elements were determined by pressurized acid digestion / inductively coupled plasma atomic emission spectrometry. The homogeneity was consistent with another homogeneity check for 12 bottles by pressurized acid digestion / inductively coupled plasma atomic emission spectrometry. The homogeneities of main constituents and non-metal elements were evaluated from the repeatabilities of quantitative analysis. The homogeneity of each element is reflected in the uncertainty of the certified value and the indicative

value.

#### 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. From the homogeneity, the recommended minimum sample mass for one analysis is 300 mg or more for total Si, 100 mg or more for total C, 10 mg or more for O, 80 mg or more for halogens and S, and 500 mg or more for the others.
- (2) The certified values and information values are shown as those for the dried material. The recommended drying condition is as follows:
  - ① The material should be dried for two hours at 135 °C.
  - ② The material should be stood at room temperature in a 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 carbide powder for fine ceramics was purchased. The material of 50 g each was packed in approximately 600 glass bottles.

#### Technical Information

This CRM includes oxygen impurity of 0.73 % (mass fraction).

#### 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:  
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Revision history

December 20, 2013: The expiration of this certificate was changed to “March 31, 2024” from “March 31, 2014”.

The value of oxygen impurity was moved from “Indicative value” to “Information”.

April 1, 2015: “Metrology Management Center” was renamed to “Center for Quality Management of Metrology.”