

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 8006-a  
No. +++

## Fine Alumina Powder for Fine Ceramics - Low Purity

This certified reference material (CRM) was produced in accordance with the NMIJ's management system and in compliance with ISO GUIDE 34:2000 and ISO/IEC 17025:2005. This CRM is intended for use in controlling the precision of analysis and confirming the validity of analytical methods and instruments during the quantitative determination of trace elements in alumina.

**Certified Values**

The certified values of 12 elements in this CRM are given in the table below. The drying instruction 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 %.

## Trace metals

Element	Certified value, mass fraction (mg/kg)	Expanded uncertainty, mass fraction (mg/kg)	Analytical method (*vide infra)
Ba	0.644	0.021	1), 2), 4), 5), 6)
Ca	165.5	4.7	1), 2), 4), 5), 6)
Cu	0.83	0.10	1), 2), 4), 5)
Fe	106.0	4.6	1), 2), 4), 5), 6)
Mg	322	6	1), 2), 4), 5), 6)
Mn	4.32	0.16	1), 3), 4), 5), 6)
Na	176	15	1), 4), 5)
Si	101.6	3.9	2), 4), 5)
Sr	7.88	0.15	1), 2), 4), 5), 6)
Ti	19.10	0.34	1), 2), 4), 5), 6)
Zn	6.96	0.19	1), 2), 4), 5), 6)
Zr	4.86	0.20	1), 2), 5), 6)

## \* Analytical method

1) pressurized acid digestion (hydrochloric acid) / inductively coupled plasma mass spectrometry, 2) pressurized acid digestion (hydrochloric acid) – aluminum separation / isotope dilution – inductively coupled plasma mass spectrometry, 3) pressurized acid digestion (hydrochloric acid) / furnace atomic absorption spectrometry, 4) pressurized acid digestion (hydrochloric acid) / inductively coupled plasma optical emission spectrometry, 5) pressurized acid digestion (sulfuric acid) / inductively coupled plasma optical emission spectrometry, 6) alkaline fusion / inductively coupled plasma optical emission spectrometry.

**Analysis**

Each certified value was determined as a weighted mean of multiple analytical results from the National Metrology Institute of Japan (NMIJ) and the Research Institute of Instrumentation Frontier (RIIF). The analytical method(s) were as follows:

- (1) combination of a primary method of measurement and other methods proved to be valid,
- (2) combination of more than two methods proved to be valid.

Even in the case of (2), the results obtained by only single digestion method or only single instrumental analysis method were not adopted as certified values.

**Metrological Traceability**

Each certified value was determined by multiple methods including the 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 several kinds of components in this CRM are given in the tables below. Each value following  $\pm$  in the columns of the information values is the expanded uncertainty determined with the coverage factor  $k = 2$ ; it defines an interval estimated to have a level of confidence of approximately 95%.

## Trace metals

Element	Indicative value, mass fraction (mg/kg)	Analytical method (*vide infra )
B	0.75 $\pm$ 0.13	4), 5)
Cr	3.0 $\pm$ 0.8	1), 2), 4), 5), 6)
Ga	51.5 $\pm$ 4.2	4), 5), 6)
V	4.2 $\pm$ 1.0	1), 4), 5), 6)

\* Analytical method

1) pressurized acid digestion (hydrochloric acid) / inductively coupled plasma mass spectrometry, 2) pressurized acid digestion (hydrochloric acid) – aluminum separation / isotope dilution – inductively coupled plasma mass spectrometry, 4) pressurized acid digestion (hydrochloric acid) / inductively coupled plasma optical emission spectrometry, 5) pressurized acid digestion (sulfuric acid) / inductively coupled plasma optical emission spectrometry, 6) alkaline fusion / inductively coupled plasma optical emission spectrometry.

## Trace non-metals

Element	Indicative value, mass fraction (mg/kg)	Analytical method
F	19.8 $\pm$ 0.9	Pyrohydrolysis / ion-chromatography
Cl	344 $\pm$ 9	Pyrohydrolysis / ion-chromatography

## Loss by strong heat

	Indicative value, mass fraction (%)	Analytical method
Loss by strong heat	0.176 $\pm$ 0.007	Strong heat for 60 min at 1100 °C (the method of JIS R 1649:2002)

**Expiration of Certification**

The certification of this CRM is valid until March 31, 2020, provided that the CRM 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. 50 g in net volume is kept in a glass bottle.

**Homogeneity**

The homogeneities of the trace metal elements in the CRM were determined by analyzing 16 bottles selected from approximately 400 bottles. The elements were determined by pressurized acid digestion (hydrochloric acid) / inductively coupled plasma optical emission spectrometry. 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 stored at a temperature between 5 °C and 35 °C, and away from a high humidity and direct sunlight.

### **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 1 g or more for loss by strong heat, 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 condition 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 alumina powder for fine ceramics was purchased. The material was mixed in a 50-L polyethylene (PE) drum after sieved with a standard nylon sieve of eyes difference 1mm; then after sieved again, the material remaining on the sieve was put in a clean PE bag and crushed by hands. The whole material was sieved again. The process of sieving and crushing was carried out repeatedly. The material passing the sieve, more than 90 % of the total, was homogenized by mixing in the PE drum. The material of 50 g each was packed in approximately 400 glass bottles.

### **NMIJ Analysts**

The technical manager and the production manager is A. Hioki, and the analysts are H. Morikawa, N. Nonose, A. Hioki, A. Tsuge and Y. Uwamino.

### **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, 2015

Ryoji Chubachi  
President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:  
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National Metrology Institute of Japan,  
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Revision history

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

Sample