National Institute of Advanced Industrial Science and Technology

# National Metrology Institute of Japan

## Reference Material Certificate

NMIJ CRM 5804-a
No. +++

National Institute of Advanced Industrial Science and Technology
AIST

## Isotropic Graphite for Thermal Diffusivity Measurement

This certified reference material (CRM) was produced in accordance with NMIJ's management system, and in compliance with ISO GUIDE 34:2000 and ISO/IEC 17025:2005. This CRM is intended for use in the calibration or confirming the validity of instruments for thermal diffusivity measurements.

**Certified Values**

The certified values for thermal diffusivity and their relative expanded uncertainties at several temperature points of this CRM are given in the following table. The quoted 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 %.

| Temperature | Certified value | Relative expanded uncertainty |
|---|---|---|
| $T$ (K) | $\alpha$ ($m^2s^{-1}$) | $U$ (%) |
| 300 | $1.00 \times 10^{-4}$ | 6 |
| 400 | $6.35 \times 10^{-5}$ | 6 |
| 500 | $4.65 \times 10^{-5}$ | 5 |
| 600 | $3.68 \times 10^{-5}$ | 5 |
| 700 | $3.05 \times 10^{-5}$ | 5 |
| 800 | $2.62 \times 10^{-5}$ | 5 |
| 900 | $2.30 \times 10^{-5}$ | 5 |
| 1000 | $2.06 \times 10^{-5}$ | 5 |
| 1100 | $1.87 \times 10^{-5}$ | 5 |
| 1200 | $1.71 \times 10^{-5}$ | 5 |
| 1300 | $1.59 \times 10^{-5}$ | 6 |
| 1400 | $1.48 \times 10^{-5}$ | 6 |
| 1500 | $1.39 \times 10^{-5}$ | 7 |

The certified values of thermal diffusivity for 300 K $\leq T \leq$ 1500 K are obtained using the following equation:

$$\alpha/(\mathrm{m^2s^{-1}}) = -3.692 \times 10^{-5} + 3.964 \times 10^{-5} \cdot \exp\left(\frac{3.719 \times 10^2}{(T/\mathrm{K})}\right).$$

Here, $\alpha$ /($m^2s^{-1}$) is thermal diffusivity and $T$/K is temperature.

The following values are obtained for relative expanded uncertainty:

$U$ = 6 % (300 K $\leq T \leq$ 440 K)
$U$ = 5 % (440 K $< T \leq$ 1200 K)
$U$ = 6 % (1200 K $< T \leq$ 1420 K)
$U$ = 7 % (1420 K $< T \leq$ 1500 K)

**Analysis**

The certified values (thermal diffusivity) were determined using the laser flash method.

Sixty specimen sets of the CRM were made from one cubic block of isotropic graphite. The set consisted of four disk-shaped specimens with a diameter of 10 mm and thicknesses of 1.4 mm, 2.0 mm, 2.8 mm, and 4.0 mm. Six sets (24 specimens) were sampled for determining the thermal diffusivity of the CRM. Thermal diffusivities at 300 K, 523 K, 773 K, 1023 K, 1273 K, and 1523 K were measured for each specimen. The measurements at 300 K were carried out in air. Above 300 K, the measurements were carried out at the setting temperature in vacuum (<1.0 Pa) by using an electric furnace. Thermal diffusivity of the CRM as a function of temperature was determined by a least squares fit for all measured thermal diffusivity values of the 24 specimens. Temperature rise curves analyzed at NMIJ were obtained from measurements performed using "CFP32 for Windows." It is software developed by NMIJ and is used for the analysis of data obtained by the laser flash method. The analysis is based on a theoretical function that takes into account the radiation heat loss. The apparent thermal diffusivity value was calculated from a temperature rise curve. Laser flash measurements were carried out using five levels of the pulsed heating energy at a steady temperature. The intrinsic thermal diffusivity at the steady temperature was determined as a value extrapolated to zero pulsed heating energy in the plot of apparent thermal diffusivity versus the maximum amplitude of temperature rise curve which depends on the pulsed heating energy. The thermal diffusivity of the CRM was calculated using the specimen thickness measured by a linear gauge at room temperature.

The relative expanded uncertainty was estimated from the uncertainty components derived from the thermal diffusivity measurements, and from the determination of the certified values as a function of temperature and homogeneity.

**Metrological Traceability**

Thermal diffusivity, derived from length, time, and temperature, was determined using a primary measurement system for thermal diffusivity at NMIJ that was calibrated using block gauges, a function generator, and an R-type thermocouple. These instruments were calibrated to be traceable to the International System of Units (SI), and thus, the certified values (thermal diffusivity) were also traceable to SI.

**Expiration of Certification**

The certification of this CRM is valid until March 31, 2020, provided that the material remains unopened and is stored in accordance with the instructions given in this certificate.

**Sample Form**

This CRM was in the form of disks with a diameter of 10 mm and thicknesses of 1.4 mm, 2.0 mm, 2.8 mm, and 4.0 mm; further, the CRM was stored in a plastic case.

**Homogeneity**

The homogeneity of this CRM was determined by an analysis of variance in the thermal diffusivity values at 300 K for specimens of six sampled sets. The homogeneity is reflected in the uncertainty of the certified value.

**Precautions for Storage**

This CRM should be stored in an environment with a temperature of 23 °C ± 10 °C and a relative humidity of 50% or less.

**Instructions for Use**

This CRM should be used only for testing and experimental studies. Further, it should be used in vacuum or under an inert gas flow (Ar, etc.) above room temperature.

**Precautions for Handling**

Handling of the CRM is similar to that of solid-state graphite. This CRM should be handled according to the instructions given in the safety data sheet (SDS).

**Preparation Method**

CRM specimens were prepared from a cubic block, with sides of 15 cm, of IG-110 produced by Toyo Tanso Ltd. IG-110 is one

of the grades of isotropic graphite.

**Information**

This CRM does not deteriorate even during a ten-fold temperature rise from 300 K to 1500 K or during an accumulated exposure of 40 h to high temperatures above 1073 K.

Reactivity to typical heat-resistant materials: This CRM does not undergo any severe reaction with $Al_2O_3$ and BN in the temperature range from room temperature to 1500 K and in vacuum or under an inert gas flow. Isotropic graphite sometimes smears sample holders with soot above 1000 K because of sublimation.

If you want to change the shape of this CRM, please handle it carefully and keep it crack-free during the machining process. After the machining process, the CRM specimen should be cleaned and dried. It is necessary to anneal the specimen for more than 3 h at 1073 K in vacuum or under an inert gas flow.

**NMIJ Analysts**

For this CRM, the technical manager is N. Yamada, and the production manager and analyst is M. Akoshima.

**Technical Information**

Customer registration on the NMIJ Website (given below) will facilitate notification of any revision of the information given above. 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.

December 22, 2010

Tamotsu Nomakuchi
President
National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:
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Note: This certificate is a translation of the original Japanese certificate and is not an official document.

Supplementary note: This CRM was supplied as NMIJ RM 1201-a until Dec. 2010. Supply as NMIJ CRM 5804-a has started since Feb. 2011.
Revision history
March 19, 2014 The expiration of this certificate was changed to “March 31, 2020” from “March 31, 2015.”