National Institute of Advanced Industrial Science and Technology

# National Metrology Institute of Japan



**Reference Material Report** 

NMIJ RM 1401-a No +++



Thermal Conductivity Reference Material (Isotropic Graphite)

This reference material (RM) is produced in accordance with the NMIJ's management system and is in compliance with ISO 17034 and ISO/IEC 17025. This RM is intended for use in calibration of thermal conductivity analyzers. It is also intended to be used for validation of analytical techniques and instruments.

# **Indicative Values**

The indicative values (thermal conductivity  $\lambda$ ) of this RM at typical temperatures and its uncertainties are given in the table below. The uncertainty of the indicativevalues is the relative expanded uncertainty obtained by multiplying the combined standard uncertainty by a coverage factor (*k*) of 2, and it is the half-width of an interval estimated to have a level of confidence of approximately 95 %.

Temperature	Indicative value	Relative expanded	
<i>T</i> (K)	λ ( W m <sup>-1</sup> K <sup>-1</sup> )	uncertainty	
		$U_{\lambda}(\%)$	
300	126	9.8	
400	110	8.4	
500	99.4	7.7	
600	90.5	7.4	
700	83.1	7.5	
80 <mark>0</mark>	76.5	7.6	
900	70.6	7.7	

The thermal conductivity  $\lambda$  (Wm<sup>-1</sup>K<sup>-1</sup>) in the table is calculated by using the following equation:

$$\lambda = \alpha \times (c_{\rm p} \times 1000) \times \rho$$

See the Reference Information given in this report for the thermal diffusivity  $\alpha$  (m<sup>2</sup>s<sup>-1</sup>), the specific heat capacity  $c_p$ (J K<sup>-1</sup> g<sup>-1</sup>) and the bulk density  $\rho$ (kg m<sup>-3</sup>) in the following page.

# Analysis

The indicative values (thermal conductivity  $\lambda$ ) of this RM is calculated as a product of the thermal diffusivity  $\alpha$ , the specific heat capacity  $c_p$ , and the bulk density  $\rho$ . The values of thermal diffusivity  $\alpha$ , specific heat capacity  $c_p$  and bulk density  $\rho$  are obtained using the methods described below. (See [Technical Information] for their values.)

The thermal diffusivity  $\alpha$  was measured by the laser flash method. Five samples for the thermal diffusivity evaluation (with a diameter of 10 mm and with a thickness of 1.0 mm, 1.4 mm, 2.0 mm, 2.8 mm and 4.0 mm) were prepared from each of ten isotropic graphite rods (with a diameter of 10 mm and with a length of 100 mm each) for distribution, which were examined to determine the value of thermal diffusivity. To be more specific, the homogeneity was first evaluated based on the room temperature measurement results for all the samples, and then by using all the sample of 2.0 mm in thickness and a pair composed of samples with different thicknesses, thermal diffusivity was measured at room temperature, about 500 K, 700 K and 900 K. Based on the all measurements, the equation of temperature dependence was obtained through the least squares method.

# Date of Shipment: Xxxxx XX, 20XX

The specific heat capacity  $c_p$  was measured by the Differential Scanning Calorimetry (the DSC method). A sample for the specific heat capacity evaluation (with a diameter of 5 mm and with a thickness of 1.0 mm) was prepared from each of ten rods for distribution, which was measured at about 360 K, 550 K, 740 K and 930 K to determine the value of specific heat capacity. Based on the all measurements, the equation of temperature dependence was obtained through the least squares method.

The bulk density  $\rho$  was measured at room temperature by using the method stipulated in JIS R 7222. Samples for the bulk density evaluation (with a diameter of 10 mm and with a thickness of 10 mm) were prepared from each of ten rods for distribution, which were evaluated to determine the value of bulk density.

The tests to assign values were performed under the following conditions:

Range of temperatures: 300 K to 900 K

Ambience: the thermal diffusivity: in the air ambience (at room temperature) and in vacuum (at less than 1.0 Pa) (higher than room temperature),

the specific heat capacity: in the nitrogen gas ambience

#### **Metrological Traceability**

The thermal diffusivity  $\alpha$  of this RM is determined from specimen thickness and heat diffusion time measured by the laser flash thermal diffusivity measurement system, which was calibrated by using block gauges, a function generator, and an R-type thermocouple, which were calibrated to be traceable to the International System of Units (SI).

The specific heat capacity  $c_p$ , which is also the reference values of this RM, is determined by a DSC using various certified reference materials and a calibrated electronic balance traceable to the SI.

The bulk density  $\rho$ , which is the third reference values of this RM, is calculated from size measured with a liner gauge calibrated by block gauges and mass measured by a calibrated electronic balance. Therefore, the thermal conductivity  $\lambda$ , the indicative value of this RM, which is obtained as a product of these three, are traceable to the SI.

#### **Expiration of Report**

This report is valid from the date of shipment to March 31, 2030, provided that the material is stored in accordance with the instructions given in this report.

#### **Description of the Material**

This RM consists of a pair of its black disc-shaped samples packaged in a plastic container: one with a diameter of 10 mm and a thickness of 1.0 mm and the other with a diameter of 10 mm and a thickness of 2.0 mm.

#### Instructions for Storage

This RM should be stored at a temperature of 23  $^{\circ}C \pm 5 ^{\circ}C$  and at a relative humidity of 50 % or less.

#### Instructions for Use

The information on the use of this **RM** is given below:

- This RM is to be used only for testing and experimental studies.
- When this RM is used at a temperature above room temperature, it must be kept in the vacuum or non-oxidizing ambience. At a temperature of 800 °C or higher, a slight amount of clouding and/or black powder was sometimes observed around the position to hold a sample of this RM, which was considered to be attributed to sublimation.
- It is not available for use when this CRM is given some damages, for example, cracks and strain.
- It is not available for use when some substances is absorbed or attached in the RM because of change of thermal diffusivity.

### **Precautions for Handling**

The procedure to handle common solid graphite should be applied. Refer to the safety data sheet (SDS) on this RM before use.

#### Preparation

The base material of this RM was ten rods of the commercially-available isotopic graphite (IG-110 manufactured by Toyo Tanso,

Co., Ltd.). Each rod was 10 mm in diameter and 100 mm in length. From these rods, disc-shaped pieces with a diameter of 10 mm and a thickness of 1.0 mm and 2.0 mm were prepared by means of machining. The pieces were annealed at a temperature of 800 °C or higher with argon gas being fed for three hours or more at NMIJ.

#### **Technical Information**

The reference values and their uncertainties of the thermal diffusivity  $\alpha$  and the specific heat capacity  $c_p$  used to calculate the indicative value of thermal conductivity of this RM at typical temperature are given in the table below. The uncertainties of the reference values,  $U_{\alpha}$  and  $U_{cp}$ , are the relative expanded uncertainty obtained by multiplying the combined relative standard uncertainty by a coverage factor (*k*) of 2, and it is the half-width of an interval estimated to have a level of confidence of approximately 95 %.

Temperature	Thermal diffusivity		Specific heat capacity		
Т	α	$U_{lpha}$	<i>C</i> <sub>p</sub>	$U_{cp}$	
(K)	$(m^2s^{-1})$	(%)	(J K <sup>-1</sup> g <sup>-1</sup> )	(%)	
300	9.68×10 <sup>-5</sup>	8.8	0.731	4.3	
400	6.21×10 <sup>-5</sup>	7.5	0.999	3.7	
500	4.57×10 <sup>-5</sup>	6.9	1.220	3.3	
600	3.63×10 <sup>-5</sup>	6.7	1.400	3.1	
700	3.02×10 <sup>-5</sup>	6.8	1.542	3.1	
800	2.60×10 <sup>-5</sup>	6.8	1.650	3.3	
900	2.29×10 <sup>-5</sup>	6.8	1.729	3.7	

The thermal diffusivity  $\alpha$  and the specific heat capacity  $_{\rm O}$  in the table were calculated by using the following equations:

$$\alpha/(\mathrm{m}^{2}\mathrm{s}^{-1}) = -3.795 \times 10^{-5} + 4.091 \times 10^{-5} \cdot \exp\left(\frac{3.576 \times 10^{2}}{(T/K)}\right)$$
  
$$c_{\mathrm{p}} = -0.39119 + 4.6281 \times 10^{-3} (T/\mathrm{K}) - 3.1676 \times 10^{-6} (T/\mathrm{K})^{2} + 7.1481 \times 10^{-10} (T/\mathrm{K})^{3}$$

Where, *T* is the temperature. The applicable temperature range is 300 K  $\leq T \leq$  900 K. The bulk density  $\rho$  for calculation of thermal conductivity in above temperature range was given as 1782 kg m<sup>-3</sup> (with uncertainty of 13.2 kg m<sup>-3</sup>(k=2)) which was determined at room temperature.

It has been demonstrated that the thermal diffusivity and the specific heat capacity of this RM remain unchanged within the range of the uncertainties when the heat cycle between room temperature and 800 °C is repeated up to ten times or when the cumulated duration of exposure to high temperature environment (up to 800 °C) is up to 60 hours. Accordingly, the stability of the thermal conductivity of this RM, which is a product of the thermal diffusivity and the specific heat capacity, is also demonstrated under the same conditions. Within the temperature range from room temperature to maximum allowable temperature for use, no reactions were observed between this RM and  $\alpha$ -alumina (Al<sub>2</sub>O<sub>3</sub>) or boron nitride (BN) in its specified ambience of use.

#### NMIJ Analysts

The technical manager for this RM is YAMADA N. the production manager is AKOSHIMA M., and the analyst is ABE H.

#### Information

If substantive technical changes occur that affect the value assignment before the expiration of this report, NMIJ will notify the registered customer. Customer registration on the NMIJ Website (given below) will facilitate notification. Technical reports regarding this RM can be obtained from the contact details given below.

#### **Reproduction of Report**

In reproducing this report, it should be clearly indicated that the document is a copy.

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April 1, 2020

ISHIMURA Kazuhiko President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this RM, 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; https://unit.aist.go.jp/nmij/english/refmate/

# Revision history

March 2, 2014: The limit of validity of the report was extended from "March 31, 2015" to "March 31, 2020."
April 1, 2015: "Metrology Management Center" was renamed to "Center for Quality Management of Metrology."
February 20, 2019: The limit of validity of the report was extended from "March 31, 2020" to "March 31, 2025."
May 30, 2025: The limit of validity of the report was extended from "March 31, 2025" to "March 31, 2030."

