

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 5806-a

No. +++



Single Crystal of Silicon for Specific Heat Capacity Measurements

(at Cryogenic Temperature)

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 can be used to control the precision of analysis for Differential Scanning Calorimeter (DSC) or confirm the validity of measurement performance check for calorimeter.

**Certified Value**

The certified value of specific heat capacity of this CRM is given in the table 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 %.

Temperature, $T / K$	Certified value, $c_p / J K^{-1} g^{-1}$	Expanded uncertainty, $U / J K^{-1} g^{-1}$	Temperature, $T / K$	Certified value, $c_p / J K^{-1} g^{-1}$	Expanded uncertainty, $U / J K^{-1} g^{-1}$
50	0.0786	0.0032	220	0.5970	0.0078
60	0.1146	0.0035	230	0.6152	0.0079
70	0.1510	0.0039	240	0.6322	0.0080
80	0.1870	0.0042	250	0.6480	0.0080
90	0.2227	0.0046	260	0.6627	0.0081
100	0.2583	0.0049	270	0.6763	0.0081
110	0.2934	0.0052	273.15	0.6805	0.0081
120	0.3280	0.0056	280	0.6891	0.0081
130	0.3617	0.0059	290	0.7009	0.0081
140	0.3942	0.0062	293.15	0.7045	0.0081
150	0.4253	0.0065	300	0.7119	0.0081
160	0.4549	0.0067	310	0.7222	0.0081
170	0.4828	0.0069	320	0.7317	0.0081
180	0.5090	0.0072	330	0.7406	0.0081
190	0.5334	0.0074	340	0.7489	0.0081
200	0.5562	0.0075	350	0.7568	0.0081
210	0.5774	0.0077			

The certified value ( $c_p$ ) and expanded uncertainty ( $U$ ) of this CRM are given in the following formulas,

at  $50\text{ K} \leq T < 100\text{ K}$ ,

$$c_p / \text{JK}^{-1} \text{g}^{-1} = 4.0206 \times 10^{-2} - 5.8826 \times 10^{-3} \cdot (T/\text{K}) + 2.4772 \times 10^{-4} \cdot (T/\text{K})^2 - 3.1655 \times 10^{-6} \cdot (T/\text{K})^3 \\ + 1.9893 \times 10^{-8} \cdot (T/\text{K})^4 - 4.9465 \times 10^{-11} \cdot (T/\text{K})^5$$

at  $100\text{ K} \leq T < 200\text{ K}$ ,

$$c_p / \text{JK}^{-1} \text{g}^{-1} = -4.0837 \times 10^{-2} + 1.7123 \times 10^{-3} \cdot (T/\text{K}) + 2.0234 \times 10^{-5} \cdot (T/\text{K})^2 - 7.1941 \times 10^{-8} \cdot (T/\text{K})^3 \\ - 6.3602 \times 10^{-11} \cdot (T/\text{K})^4 + 3.8280 \times 10^{-13} \cdot (T/\text{K})^5$$

at  $200\text{ K} \leq T \leq 350\text{ K}$ ,

$$c_p / \text{JK}^{-1} \text{g}^{-1} = -0.81477 + 1.6542 \times 10^{-2} \cdot (T/\text{K}) - 8.6727 \times 10^{-5} \cdot (T/\text{K})^2 + 2.6977 \times 10^{-7} \cdot (T/\text{K})^3 \\ - 4.5515 \times 10^{-10} \cdot (T/\text{K})^4 + 3.1789 \times 10^{-13} \cdot (T/\text{K})^5$$

at  $50\text{ K} \leq T \leq 350\text{ K}$ ,

$$U / \text{JK}^{-1} \text{g}^{-1} = 0.0017509 + 2.1144 \times 10^{-5} \cdot (T/\text{K}) + 1.9668 \times 10^{-7} \cdot (T/\text{K})^2 - 1.0623 \times 10^{-9} \cdot (T/\text{K})^3 \\ + 1.3595 \times 10^{-12} \cdot (T/\text{K})^4$$

### Analysis

The certified value of this CRM was determined by the following analytical methods:

- (1) Specific heat capacity of a sample was determined by heat capacity measurement by an adiabatic calorimeter applying a pulse-tube cryocooler in the temperature range from 50 K to 350 K and the mass measurement by a balance.
- (2) The certified values were decided as a function of the temperature by obtaining the regression curve by the least-squares method.
- (3) The combined standard uncertainty was determined by composition of standard uncertainty about the measurement, homogeneity and stability of the sample, and the regression curve decision by the least-squares method.

### Metrological Traceability

The certified value was determined by the adiabatic calorimeter based on the measurements, namely temperature (a platinum resistance thermometer), voltage (a digital multimeter), resistance (a standard resistance), heating time (a counter), and mass (an electronic balance), for which traceability is secured. The certified value is traceable to the International System of Units (SI).

### Expiration of Certification

This certificate is valid from the date of shipment to March 31, 2026, provided that this CRM is stored in accordance with the instructions given in this certificate.

### Description of the material

The form of this CRM is a disk with a diameter of 5 mm and a thickness of 1 mm and kept in a plastic case.

### Homogeneity

The homogeneity of this CRM was determined by analysis of variance. The specimens were cut from four different positions in a single-crystalline silicon ingot. This uncertainty of homogeneity has been incorporated in the uncertainty of the certified value.

### Instructions for Storage

This CRM is recommended to be preserved in a desiccator.

### Instructions for Use

- This CRM is for laboratory use only. Additionally, it is applied to general handling of the solid silicon.
- Temperature
  - Avoid a rapid temperature change that a crack is caused in the CRM.
  - Avoid heating to 350 K or more.

- Processing of a test piece
  - It is possible to process the CRM into a suitable shape.
  - During processing, neither a crack nor a distortion is caused in the CRM.
- Polish the contact surface with a sample crucible.

### Precautions for Handling

Wear suitable protective gloves when handling. Refer to the safety data sheet (SDS) on this CRM before use.

### Preparation

This CRM was cut from a single-crystalline silicon ingot made by Floating zone method. The form is a disk with a diameter of 5 mm and a thickness of 1 mm.

### Technical Information

This CRM does not degrade even after going through 100 thermal cycles from 77 K to the room temperature.

### NMIJ Analysts

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

### Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the registered customers. 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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National Metrology Institute of Japan,  
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Phone: +81-29-861-4059; Fax: +81-29-861-4009, <https://unit.aist.go.jp/nmij/english/refmate/>

#### Revision history

Nov. 12, 2014: The limit of validity of the certificate was extended to “March 31, 2021” from “March 31, 2016”.

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

Dec. 3, 2020: The limit of validity of the certificate was extended to “March 31, 2026” from “March 31, 2021”.