

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 5715-a

No. +++



Carbon black (Nitrogen Specific Volume Adsorbed – BET 20)

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 is a carbon black characterized for specific volume of adsorbed nitrogen and for specific surface area based on the multipoint Brunauer-Emmett-Teller (BET) method. It is intended for use in the quality control of the nitrogen adsorption method and the validation of its measurement methods and instruments.

#### Certified Values

The certified values of specific volume of adsorbed nitrogen and specific surface area for this CRM are given in the tables below. The uncertainty of each certified value is the 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 of confidence estimated to have a level of confidence of approximately 95 %. Relative pressure is the ratio of the equilibrium adsorption pressure to the saturation vapor pressure of nitrogen at 77 K.

Specific volume of adsorbed nitrogen\*

Relative pressure (Pa/Pa)	Certified value Specific volume of adsorbed nitrogen (cm <sup>3</sup> /g)	Expanded uncertainty Specific volume of adsorbed nitrogen (cm <sup>3</sup> /g)	Relative pressure (Pa/Pa)	Certified value Specific volume of adsorbed nitrogen (cm <sup>3</sup> /g)	Expanded uncertainty Specific volume of adsorbed nitrogen (cm <sup>3</sup> /g)
0.050	3.90	0.11	0.400	6.54	0.25
0.075	4.14	0.11	0.450	6.94	0.28
0.100	4.35	0.12	0.500	7.36	0.30
0.125	4.53	0.12	0.550	7.80	0.32
0.150	4.71	0.13	0.600	8.27	0.36
0.175	4.88	0.14	0.650	8.82	0.40
0.200	5.06	0.14	0.700	9.45	0.48
0.225	5.23	0.15	0.750	10.24	0.55
0.250	5.41	0.16	0.800	11.27	0.62
0.275	5.60	0.18	0.850	12.76	0.67
0.300	5.78	0.19	0.900	15.5	1.8
0.350	6.16	0.22			

\*Specific volume of adsorbed nitrogen in the table is volumetric equivalent of specific amount adsorbed (mol/g) expressed as gas under standard conditions of temperature and pressure (STP: 101 325 Pa and 273.15 K). The specific volume of adsorbed nitrogen shown in the table can be converted to the specific amount of nitrogen adsorbed (mol/g) by being multiplied by  $4.462 \times 10^{-5}$  mol/cm<sup>3</sup>.

## Specific Surface Area

Certified value Specific surface area (m <sup>2</sup> /g)	Expanded uncertainty Specific surface area (m <sup>2</sup> /g)
18.0	1.2

**Analysis**

The certified values of specific volume of adsorbed nitrogen were determined by the static volumetric method (ISO 9277:2010) at the temperature of liquid nitrogen, 77 K. The certified value of specific surface area was determined by the multipoint BET method using the measured values of specific volume of adsorbed nitrogen in the relative pressure range of 0.05 to 0.25 in accordance with ISO 9277:2010. Cross-sectional area of nitrogen molecule of 0.162 nm<sup>2</sup> was used.

**Metrological Traceability**

The certified values of specific volume of adsorbed nitrogen were determined using a measurement system calibrated with a pressure gauge, a thermometer, weights, and gauge blocks that were calibrated in the Japan Calibration Service System (JCSS). Therefore, these certified values are traceable to the International System of Units (SI). The certified value of specific surface area was determined by the multipoint BET method based on the SI-traceable measurement values of specific volume of adsorbed nitrogen.

**Indicative Value**

Specific volume of adsorbed nitrogen at the relative pressure of 0.95 and its expanded uncertainty (coverage factor (*k*) of 2) are given in the table below. Although this value was determined by the same method as those at the other relative pressures, it is provided as an indicative value because the uncertainty is substantially large.

Relative pressure (Pa/Pa)	Indicative value Specific volume of adsorbed nitrogen (cm <sup>3</sup> /g)	Expanded uncertainty Specific volume of adsorbed nitrogen (cm <sup>3</sup> /g)
0.95	23.9	4.3

**Expiration of Certification**

This certificate is valid for one year from the date of shipment, provided that this CRM is stored in accordance with the instructions given in this certificate.

**Description of the material**

This CRM is in the form of black granulated powder. Approximately 13 g of this CRM in net weight is kept in an amber glass bottle.

**Instructions for Storage**

This CRM should be stored at temperature of 5 °C to 35 °C. Its container should be tightly closed and protected from direct daylight. This CRM should be stored in clean ambient air once a container is opened.

**Instructions for Use**

More than 0.5 g of this CRM should be sampled for each measurement to maintain the homogeneity. Care should be taken to avoid breathing fine particles and crushing granules. As a pre-treatment, this CRM should be heated at 300 °C for 2 hours at an absolute pressure of 1 Pa to 5 Pa to complete outgassing. A sample should not be used repeatedly. Specific surface area should be determined in accordance with the procedure of the multipoint BET method described in ISO 9277:2010 by measuring specific volume of adsorbed nitrogen at evenly-spaced four or more points at a relative pressure of 0.05 to 0.25s. The

cross-sectional area of 0.162 nm<sup>2</sup> for nitrogen molecule should be used.

### Precautions for Handling

Care should be taken to avoid scattering the powder. If necessary, a mask, gloves, and other personal protective equipment should be used. Refer to the safety data sheet (SDS) on this CRM before use.

### Preparation

This CRM was prepared by the following steps: 1) commercially-available ungraphitized carbon black was sieved, 2) the sieved carbon black was heated by microwave to carbonize unburned petroleum components, and 3) the carbon black was split so as not to cause segregation and bottled. The sieving process was performed by National Institute of Advanced Industrial Science and Technology. The microwave heating process and the dividing and bottling process were undertaken by STU Co., Ltd and Seishin Enterprise Co., Ltd, respectively.

### NMIJ Analysts

The technical manager for this CRM is SAKURAI Hiromu and the production manager and analyst is MIZUNO Kohei.

### 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.

February 25, 2021

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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