# National Institute of Advanced Industrial Science and Technology

# National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 5714-a No. +++



Carbon Black (Nitrogen Specific Volume Adsorbed - BET100)

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 values 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.

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	Certified value	Expanded		Certified value	Expanded
		uncertainty			uncertainty
Polativo	Specific volume	Specific volume	Polativo	Specific volume	Specific volume
prossure	of adsorbed	of adsorbed	Drossuro	of adsorbed	of adsorbed
(Pa/Pa)	nitrogen	nitrogen	(Po/Po)	nitrogen	nitrogen
(га/га)	(cm³/g)	(cm <sup>3</sup> /g)	(Fa/Fa)	(cm³/g)	(cm³/g)
0.050	24.4	1.3	0.400	39.7	1.6
0.075	25.63	0.53	0.450	42.1	1.8
0.100	26 <mark>.7</mark> 0	0.59	0.500	44.6	2.0
0.125	27.73	0.66	0.550	47.2	2.2
0.150	28.74	0.72	0.600	50.2	2.5
0.175	29.76	0.79	0.650	53.6	2.8
0.200	30.79	0.87	0.700	57.6	3.1
0.225	31.85	0.94	0.750	62.8	3.5
0.250	32.9	1.0	0.800	70.0	4.2
0.275	34.0	1.1	0.850	81.0	5.5
0.300	35.2	1.2	0.900	105	11
0.350	37.4	1.4			

# Specific Volume of Adsorbed Nitrogen\*

\*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>.

Certified value	Expanded uncertainty		
Specific surface area	Specific surface area		
(m²/g)	(m²/g)		
110.0	7.3		

#### Specific Surface Area

#### 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 range of 0.05 to 0.25 in accordance with ISO 9277:2010. Cross-sectional area of nitrogen molecule of  $0.162 \text{ nm}^2$  was used. The specific surface area determined from the certified values of specific volume of adsorbed nitrogen is  $109.0 \text{ m}^2/\text{g}$ .

# 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 by 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.

# **Expiration of Certification**

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

#### **Description of the material**

This CRM is in the form of a black granulated powder of carbon black. Approximately 12 g of this CRM in net weight is kept in an amber glass bottle.

#### **Instructions for Storage**

This CRM should be stored at temperature between 5 °C and 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 0.1 g of the material should be sampled for each measurement with regard to 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 3 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 0.25. The cross-sectional area of 0.162 nm<sup>2</sup> for nitrogen molecule should be used.

# Precautions for Handling

Care must be taken to prevent scattering the powder. If necessary, wear a mask, gloves, and other protective equipment during handling. Refer to the safety data sheet (SDS) on this CRM before use.

#### Preparation

Commercially-available ungraphitized carbon black was sieved, split so as not to cause segregation, and bottled.. The sieving process was performed by National Institute of Advanced Industrial Science and Technology. Splitting and bottling processes were undertaken by Seishin Enterprise Co., Ltd.

# NMIJ Analysts

The technical manager for this CRM is SAKURAI H., the production manager and analyst is MIZUNO K.

# 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: 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; Fax: +81-29-861-4009, https://unit.aist.go.jp/nmij/english/refmate/

Revision history

January 29, 2019: The certified values and the expanded uncertainties were revised.

