

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Report

NMIJ RM 5713-a
No. +++

Titanium(IV) Oxide Nanoparticles
(specific surface area 76 m²/g, small particle size, surface modified with isobutyl groups)

This reference material (RM) is produced in accordance with the NMIJ's management system and in compliance with ISO 17034 and ISO/IEC 17025. This RM is intended for the calibration of instruments, and validation of analytical techniques used for the determination of specific surface area (multipoint BET method based on nitrogen gas adsorption at 77 K)

Indicative Value

The indicative value of this RM is given in the table below. The uncertainty of the indicative 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 estimated to have a level of confidence of approximately 95 %.

Substance	Indicative value Specific surface area (m ² /g)	Expanded uncertainty Specific surface area (m ² /g)
Titanium(IV) oxide rutile type surface modified with isobutyl groups (hydrophobic)	75.7	2.6

Analysis

The indicative value was determined by the multipoint BET method in accordance with JIS Z 8830:2013 (ISO 9277:2010) on the basis of volumetric measurement of nitrogen gas adsorption at 77 K. Prior to the measurement, degassing at 120 °C for 20 minutes in a vacuum was carried out. Data analysis was performed using four or more data points in a relative pressure range 0.1 to 0.2 and a molecular cross-sectional area of 0.162 nm². The indicative value was validated using certified reference material BAM-P105.

Expiration of Report

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

Description of the material

This RM consists of white powder, which is packaged in an aluminum-laminated bag (ca. 5 g each).

Instructions for Storage

This RM shows a weak photocatalytic activity. It should be kept at a temperature between 5 °C and 35 °C and shielded from direct sunlight. This RM should be stored in clean ambient air once a container is opened.

Instructions for Use

Degassing should be performed by heating at 120 °C for 20 minutes in a vacuum less than 10 Pa. More than 0.2 g of the material should be used in terms of homogeneity.

Precautions for Handling

This RM consists of particles in the size of nanometer order. It should be handled in accordance with the government guidelines for safety of nanomaterials. Refer to the safety data sheet (SDS) on this RM before use.

Preparation

The titanium(IV) oxide nanoparticles were synthesized by a sulfuric acid method. Surface of the particles was modified with isobutyl functional group to use as an external additive of toner. Synthesis and packaging were carried out by Tayca Corporation, Japan.

Technical Information

Information about average crystallite size is shown below. The information is necessary when the RM is used as a representative test material (ISO/TS 16195:2013) in developing and validating test methods using nano-objects (for example, toxicology tests).

Average crystallite size

The average crystallite size was estimated by powder X-ray diffraction and Scherrer's equation:

$$d = \frac{K\lambda}{\beta \cos \theta}$$

where d is the average crystallite size [nm], K is the Scherrer constant (0.94), λ is the X-ray wavelength (= 0.15406 nm), β is the line broadening in the full width at half maximum (FWHM) [rad], and θ is the Bragg angle [rad]. The value of β was calculated by the difference in FWHM between the reference material and NIST SRM 640d silicon powder.

Ten bags taken from 200 bags were measured to indicate homogeneity of the average crystallite size in 2011. Mean (\bar{d}) and experimental standard deviation ($s(d)$) among the ten bags are shown below.

Bragg angle (rad)	Average crystallite size (nm)	
	Mean \bar{d}	Experimental standard deviation $s(d)$
0.2388	20.0	0.5

Two samples taken from a randomly-selected bag were measured after a year in 2012 to check long-term stability of the average crystallite size. Mean of the measurement result of the two samples is 17.5 nm, which fell within the range of $\bar{d} \pm 2s(d)$.

NMIJ Analysts

For this RM, the technical manager is SAKURAI H. The production manager is MIZUNO K. The analysts are MIZUNO K. and AZUMA Y.

Information

If substantive technical changes occur that affect the value assignment before the expiration of this report, NMIJ will notify the registered customers. 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.

April 1, 2020

ISHIMURA Kazuhiko
President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this RM, please contact:
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Revision history

April 1, 2015: "Metrology Management Center" was renamed to "Center for Quality Management of Metrology."
March 14, 2018: The indicative value and the expanded uncertainty were revised. The description in "Expiration of Certification" was changed to "one year from the date of shipment."