National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan

Reference Material Report

NMIJ RM 5712-a
No. +++

Titanium(IV) Oxide Nanoparticles
(specific surface area 57 m²/g, small particle size, surface modified with fatty acid)

This reference material (RM) was produced in accordance with the NMIJ’s management system and in compliance with ISO GUIDE 34:2009 and ISO/IEC 17025:2005. This RM is intended for use in controlling the precision of analysis and apparatus for the determination of specific surface area (multipoint BET method based on nitrogen gas adsorption at 77 K).

**Indicative Value**
The indicative value of specific surface area (multipoint BET method based on nitrogen gas adsorption at 77 K) is given in the table below. The uncertainty of the indicative 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%.

<table>
<thead>
<tr>
<th></th>
<th>Indicative value (m²/g)</th>
<th>Expanded uncertainty (m²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium(IV) oxide rutile type surface modified with fatty acid (hydrophobic)</td>
<td>56.0</td>
<td>1.7</td>
</tr>
</tbody>
</table>

**Analysis**
The indicative value was determined by 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 the material remains unopened and is stored in accordance with the instructions given in this report.

**Sample Form**
This RM is in the form of a white powder, which is packaged in an aluminum-laminated bag (ca. 10 g each).

**Homogeneity**
The homogeneity of this RM was evaluated by multipoint BET method for 10 bags taken from total 200 bags. The homogeneity of analyte is reflected in the uncertainty of the indicative value.

**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.
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 Method
The titanium(IV) oxide nanoparticles were synthesized by a sulfuric acid method. Surface of the particles was modified with fatty acid group for cosmetic applications. Synthesis and packaging were carried out by Tayca Corporation, Japan.

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), \( \lambda \) is the X-ray wavelength (= 0.15406 nm), \( \beta \) is the line broadening in the full width at half maximum (FWHM) [rad], and \( \theta \) is the Bragg angle [rad]. The value of \( \beta \) 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.

<table>
<thead>
<tr>
<th>Bragg angle (rad)</th>
<th>Average crystallite size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>Mean ( \bar{d} )</td>
</tr>
<tr>
<td>0.2392</td>
<td>24.6</td>
</tr>
</tbody>
</table>

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 23.0 nm, which fell within the range of \( \bar{d} \pm 2s(d) \).

NMIJ Analysts
For this RM, the technical manager is H. Sakurai. The production manager is K. Mizuno. The analysts are K. Mizuno and Y. Azuma.

Technical Information
Customer registration on the NMIJ Website (given below) will facilitate notification of any revisions of the information given above. 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, 2015

Ryoji Chubachi
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; Fax: +81-29-861-4009, https://www.nmij.jp/english/service/C/

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 changed. The description in “Expiration of Certification” was changed to “one year from the date of shipment.”