Certified Reference Material

BAM-P116

BET Specific Surface Area of Titanium Dioxide (Anatase)

calculated from the nitrogen adsorption isotherm at 77.3 K

Certified Value

Property		Value	U [a]	$2 \cdot s_x$ [b]	Unit
Specific Surface Area [c]	A_{BET}	325	11	20	m^2/g

[a] Uncertainty $U = k \cdot u_c$ calculated according to ISO Guide 35 and ISO/IEC Guide 98-3 with the coverage factor k = 2 (giving a level of confidence of approximately 95%). The value of the combined standard uncertainty u_c of the certified property includes both an uncertainty contribution resulting from the inter-laboratory characterization, the study of inhomogeneities, stability of the material and the uncertainty contribution due to the measurement result variations of the single instruments (mean data set).

[b] Standard deviation of the accepted data set mean (twofold value).

[c] Specific surface area calculated in a relative adsorption pressure range $0.08 \leq p/p_0 < 0.25$ as multi point BET model with minimum of five points described in ISO 9277.

End of Validity

This certificate is valid for two years from the date of shipment provided the reference material is stored under the recommended conditions.

Date of Shipment from BAM:

Material Description

A unit of the CRM BAM-P116 consists of a single glass bottle containing approximately 8 g of crystalline, pure titanium dioxide powder in the modification anatase (99.5%) with a mean particle size of about 25 µm.

Recommended Use

Prior to the measurement, outgassing of the sample is necessary. Outgassing has to be carried out in vacuum with a final pressure of < 10 Pa. Heat the sample for degassing in vacuum with a rate of about 5 K/min to 453.15 K, then hold temperature at 453.15 K for at least three hours. During sample pre-treatment, a mass loss of 10% is to be expected. Afterwards, allow the sample to cool slowly down to ambient temperature.

The adsorption branch of the N_2 isotherm must be measured at 77.3 K. Perform the analysis following the instrument manufacturer's instructions. The recommended minimum sample intake is 0.5 g.

The sample preparation station should have a separate vacuum circuit in addition to the analysis station or the preparation should be carried out at a separate heating station. For instruments with a combined vacuum system, measurements and sample preparation should not be performed together, as condensation in glass vessels can occur during sample preparation.

The certified value $2s_x$ "standard deviation of the accepted data set mean value" can be used to validate gas sorption instruments in test laboratories and to carry out monitoring that is required according to ISO/IEC 17025.
This uncertainty representing the range of accepted measurement data observed in the certification interlaboratory comparison.

Transport, Storage and Handling

CRM BAM-P116 can be shipped at ambient temperature. Upon receipt the material should be stored at a temperature below 30°C in its original tightly closed bottle. When handling the sample, the bottle should be opened as briefly as possible. Care should be taken to avoid moisture pick-up once the bottle is opened. BAM cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened bottles. The material should be used as it is from the bottle. However, before taking a sub-sample re-homogenisation by manual shaking of the closed bottle is strongly recommended.

Analytical Method

The reference material is intended for performance testing of gas sorption instruments operating by the static-volumetric method. For this purpose, the instrument must perform the nitrogen gas adsorption at 77 K.

Participating Laboratories

3P INSTRUMENTS GmbH & Co. KG, Odelzhausen (Germany)

Anton Paar QuantaTec Inc., Boynton Beach (USA)

Bundesanstalt für Materialforschung und –prüfung (BAM), Div. 1.3, Berlin (Germany)

Bundesanstalt für Materialforschung und –prüfung (BAM), Div. 5.4, Berlin (Germany)

Evonik Technology & Infrastructure GmbH, Hanau-Wolfgang (Germany)

Evonik Technology & Infrastructure GmbH, Marl (Germany)

Forschungsinstitut für Anorganische Werkstoffe -Glas/Keramik- GmbH, Höhr-Grenzhausen (Germany)

Forschungszentrum Jülich GmbH, Jülich (Germany)

Fritz-Haber-Institut der Max-Planck-Gesellschaft, Berlin (Germany)

Institute of Chemical and Engineering Sciences (ICES), Jurong Island (Singapore)

Instituto Nacional de Técnica Aeroespacial, Madrid (Spain)

Instituto Pedro Nunes, Coimbra (Portugal)

IPT - Instituto de Pesquisas Tecnológicas, São Paulo (Brazil)

Oerlikon Metco WOKA GmbH, Barchfeld-Immelborn (Germany)

Particle Testing Authority - Micromeritics GmbH, Unterschleißheim (Germany)

Particle Testing Authority - Micromeritics Instrument Corp., Norcross, GA (USA)

Universiti Teknologi PETRONAS, Seri Iskandar (Malaysia)

Ural Scientific Research Institute for Metrology (UNIIM), Yekaterinburg (Russian Federation)

Means of Accepted Data Sets

Data set no.	A_{BET} (m^2/g)
C01	323.5382
C02	335.5118
C04	323.1350
C06	330.2702
C09	332.3300
C10	306.2448
C11	325.6660
C13	303.9440

Data set no.	A_{BET} (m^2/g)
C15	313.3392
C16	318.5712
C17	328.6232
C18	328.9886
C19	322.3763
C26	332.1161
C27	331.6699
C28	330.3799

Metrological Traceability

The certified value of specific surface area based on the BET method described in ISO 9277 and is traceable to the base units of the SI via calibrated measurements of the quantities pressure, volume and mass.

Literature

A detailed technical report describing the production, characterization and the treatment of the analytical data used to certify BAM-P116 is available on request or can be downloaded from BAM website (https://rrr.bam.de).

Accepted as a BAM-CRM on February 19, 2020

Bundesanstalt für Materialforschung und -prüfung (BAM)

Dr. Silke Richter
Committee for Certification

Dr. Franziska Emmerling
Project Coordinator
Head of Division 6.3
Structure Analysis

BAM holds an accreditation as a reference material producer according to ISO 17034. This accreditation is valid only for the scope as specified in the certificate D-RM-11075-01-00. DAkkS is a signatory of the multilateral agreement (MLA) between EA, ILAC and IAF for mutual acceptance.	DAkkS Deutsche Akkreditierungsstelle D-RM-11075-01-00

This Reference Material is offered by:

Bundesanstalt für Materialforschung und -prüfung (BAM)
Richard-Willstätter-Str. 11, D-12489 Berlin, Germany

P: +49 30 8104 2061
F: +49 30 8104 72061

E-Mail: sales.crm@bam.de
Internet: www.webshop.bam.de