

Certified Reference Material

BAM-P114

BET Specific Surface Area of Titanium Dioxide (Anatase/Rutile)
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}	24.48	0.30	0.4	m ² /g

^a Expanded 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 interlaboratory 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 precision).

^b Twofold standard deviation s_x of accepted laboratory means.

^c Specific surface area A_{BET} calculated in a relative adsorption pressure range of $0.05 \leq p/p_0 < 0.3$ as multi-point BET model with a minimum of five supporting points as described in ISO 9277.

Validity of the Certificate

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 BAM-P114 consists of a single glass bottle containing approximately 18 g of crystalline titanium dioxide (TiO₂) powder consisting of a mix of the modifications anatase (56 %) and rutile (44 %) with a particle size distribution ranging from 10 to 100 µm and a pore sizes of 30 nm.

Recommended Use

Prior to the measurement, outgassing of the sample is necessary. Outgassing must be carried out in vacuum with a final pressure below 10 Pa. For degassing in vacuum, the sample is to be heated with a rate of about 5 K/min to 453.15 K. Afterwards, this temperature must be maintained for at least three hours. During sample pre-treatment, a mass loss of 1.2 % is to be expected. After this period, the sample must be allowed to cool down slowly to ambient temperature.

The adsorption branch of the N₂ isotherm must be measured at 77.3 K. The analysis should be performed according to the instrument manufacturer's instructions. The recommended sample intake is 1.5 g.

The sample preparation station should have a separate vacuum circuit in addition to the analysis station or the preparation should be carried out in 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 $2 s_x$ "Twofold standard deviation of accepted laboratory means" 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 represents the range of accepted laboratory means observed in the certification interlaboratory comparison.

Transport, Storage and Handling

BAM-P114 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 to avoid deterioration of the certified value. 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

ALFATESTlab, Cinisello Balsamo (Italy)

Bundesanstalt für Materialforschung und -prüfung (BAM), Div. 6.3, Berlin (Germany)

ChemiLytics GmbH & Co. KG, Goslar (Germany)

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

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

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

Instituto Pedro Nunes, Coimbra (Portugal)

Oerlikon Metco WOKA GmbH, Barchfeld-Immelborn (Germany)

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

TUBITAK NIM, Gebze, Kocaeli (Turkey)

Universiti Teknologi Malaysia, Johor Bahru (Malaysia)

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

Means of Accepted Interlaboratory Comparison Data Sets

	A_{BET}
Data set no.	m ² /g
P01	24.4238
P02	24.4984
P03	24.8270
P04	24.4258
P05	24.1974
P06	24.3032
P07	24.5439
P08	24.3904
P09	24.4760
P10	24.8130
P11	24.4381
P12	24.6270
P13	24.2222

Metrological Traceability

The certified specific surface area is determined according ISO 9277 and 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-P114 is available on request or can be downloaded from BAM website (<https://rrr.bam.de>).

Accepted as a BAM-CRM on August 25, 2021

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 DIN EN ISO 17034:2017. 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.



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