

Certified Reference Material

BAM-P115

Pore size parameters of nanoporous Titanium dioxide
calculated from the nitrogen sorption isotherm at 77.3 K

Certified Values

Property		Value	U^a	$2 \cdot s_x^b$	Unit
Specific Surface Area ^c	A_{BET}	147.3	2.8	7.3	m ² /g
Specific pore volume ^d	$V_{\text{p},0.99}$	0.214	0.004	0.007	cm ³ /g
Hydraulic pore diameter	$4V_{\text{p},0.99}/A_{\text{BET}}$	5.79	0.07	0.07	nm
Modal pore diameter	$D_{\text{BJH,des}}^e$	4.75	0.21	0.46	nm
Modal pore diameter	$D_{\text{BJH,ads}}^f$	5.40	0.24	0.61	nm

^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 variation in the measurement results from individual instruments (mean data set).

^b Standard deviation of accepted laboratory mean values (reported as twice the standard deviation).

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

^d Single point total pore volume according to the Gurvich rule determined from the adsorption branch of the isotherm at relative pressure $p/p_0 = 0.990$

^e Pore size at maximum of the differential pore size distribution calculated from the desorption branch of the isotherm applying the BJH model as described in ISO 15901-2

^f Pore size at maximum of the differential pore size distribution calculated from the adsorption branch of the isotherm applying the BJH model as described in ISO 15901-2

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 CRM BAM-P115 consists of a single glass bottle containing approximately 12 g of crystalline titanium dioxide powder in the modification anatase with a mean particle size of about 30 μm .

Recommended Use

Prior to the measurement, outgassing of the sample is necessary. Outgassing must be carried out in vacuum with a final pressure of < 10 Pa. For degassing in vacuum, the sample is to be heated with a rate of about 5 K/min to 453.15 K, then this temperature must be maintained for at least three hours. Afterwards, the sample to be allowed to slowly cool down to ambient temperature.

The adsorption and desorption branch of the N_2 isotherm must be measured at 77.3 K. The analysis should be performed following the instrument manufacturer's instructions. The recommended minimum sample intake is 0.8 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 certified value $2s_x$ "Standard deviation of accepted laboratory mean values" 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 measurement data observed in the certification interlaboratory comparison.

Transport, Storage and Handling

CRM BAM-P115 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 and desorption at 77 K.

Participating Laboratories

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

Anton Paar QuantaTec Inc., Boynton Beach (USA)

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

Instituto Nacional de Técnica Aeroespacial, Madrid (Spain)

Instituto Pedro Nunes, Coimbra (Portugal)

Particle Testing Authority - Micromeritics GmbH, Unterschleißheim (Germany)

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

University Industry Research Laboratory (UIRL), Johor Bahru (Malaysia)

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

Means of Accepted Data Sets

Property (x)→	A_{BET}	$V_{\text{p},0.99}$	$4V_{\text{p}}/A_{\text{BET}}$	$D_{\text{BJH(des)}}$	$D_{\text{BJH(ads)}}$
Data set no.↓	m^2/g	cm^3/g	nm	nm	nm
C01	143.77	0.2093	5.827	4.414	4.913
C04	150.17	0.2146	5.735	4.548	4.936
C06	148.62	0.2152	5.791	4.560	5.100
C07	143.75	0.2085	5.802	4.578	5.292
C08	151.05	0.2169	5.744	4.585	5.304
C10	148.67	0.2160	5.808	4.619	5.324
C11	144.73	0.2108	5.826	4.805	5.328
C13	139.20	-	5.766	4.805	5.417
C15	145.59	0.2116	5.812	4.809	5.523
C17	149.83	0.2190	5.847	4.884	5.625
C19	151.46	0.2181	5.760	4.901	5.719
C21	147.36	0.2134	5.793	5.011	5.781
C27	150.25	0.2172	5.782	5.240	5.884

Metrological Traceability

The certified values of specific surface area and porosity parameters are determined according ISO 9277 and ISO 15901-2 and are 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-P115 is available on request or can be downloaded from BAM website (<https://rrr.bam.de>).

Accepted as a BAM-CRM on July 1, 2020

Bundesanstalt für Materialforschung und -prüfung (BAM)

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Committee for Certification

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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.



This Reference Material is offered by:

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