Out of stock

BAM
Bundesanstalt für Materialforschung und -prüfung

12200 Berlin, Germany
T: +49 30 8104-0
F: +49 30 8104-7 2222

Certified Reference Material

BAM-P106

Pore size parameters of nanoporous TiO_2 calculated from the nitrogen isotherm at 77.3 K

Certified Values

Property		Value	U [a]	Unit
Specific Surface Area [b]	A_{BET}	96.6	1.7	m^2/g
Specific pore volume [c]	$V_{p,0.99}$	0.2341	0.0024	cm^3/g
Hydraulic pore diameter	$4V_{p,0.99}/A_{BET}$	9.69	0.16	nm
Modal pore diameter [d]	$D_{BJH,des}$	8.2	1.0	nm
Modal pore diameter [e]	$D_{BJH,ads}$	11.5	0.9	nm

[a] Uncertainty $U = k\,u_c$ calculated according to ISO Guide 35 with the coverage factor $k = 2$. The value of the combined standard uncertainty u_c of each certified property includes both an uncertainty contribution resulting from the interlaboratory testing and a second one taking into account possible inhomogeneities of the material.

[b] BET specific surface area calculated according to ISO 9277.

[c] Single point total pore volume according to the Gurvich rule (see e.g. Gregg and Sing 1982) determined from the adsorption branch of the isotherm at relative pressure p/p_0 = 0.990.

[d] Pore size at maximum of the differential pore size distribution calculated from the desorption branch of the isotherm applying the BJH model according to ISO 15901-2.

[e] Pore size at maximum of the differential pore size distribution calculated from the adsorption branch of the isotherm applying the BJH model according to ISO 15901-2.

Expiration 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:

CERTIFICATE

Material description

A unit of BAM-P106 consists of a single glass bottle containing approximately 15 g of crystalline nanoporous TiO_2 material.

Recommended use

The reference material is intended for checking the performance of instruments used for the determination of BET specific surface area, specific pore volume, hydraulic pore diameter and BJH most frequent pore diameters from the desorption and the adsorption branch of the nitrogen isotherm determined by the static volumetric method.

Handling

Prior to the measurement, outgassing of the sample is necessary. Outgassing has to be carried out in a vacuum. Heat the sample for degassing in a vacuum with a rate of about 5 K/min to 180 °C, then hold temperature at 180 °C for at least 3 hours. Afterwards, allow the sample to cool slowly back to ambient temperature. The final pressure should be < 0.1 Pa. The adsorption and the desorption branch of the N_2 isotherm has to be measured at 77.3 K. Perform the analysis following the instrument manufacturer's instructions. The recommended minimum sample intake is 0.8 g.

Determine the BET specific surface area in accordance with the international standard ISO 9277 using at least 5 adsorption isotherm data points in the relative pressure range $0.05 \leq p/p_0 < 0.3$ (BET evaluation range of the reference material). Use the value 0.162 nm² for the cross sectional area of nitrogen.

The single point total pore volume $V_{p,0.99}$ according to the Gurvich rule should be determined from the adsorption branch of the isotherm at relative pressure $p/p_0 = 0.99$. In the case that the very last part of the adsorption isotherm is influenced by the beginning macroscopic condensation outside the pore system (indicated by a steep increase of volume) it is recommended to determine the Gurvich single point total pore volume at a lower relative pressure ≥ 0.95 in the plateau of the isotherm.

Use the factor 0.001547 for density conversion of gaseous nitrogen at STP to liquid nitrogen at 77.3 K, i. e. $V_{p,0.99} = 0.001547 \times V_{a,0.99}$.

The relationship $4V_{p,0.99}/A_{BET}$ has to be used for the calculation of the hydraulic pore diameter.

Perform BJH calculations for the adsorption and desorption branches of the isotherm using your instrument's software package. The Halsey thickness curve should be used for the BJH calculations. Determine the modal pore diameter $D_{BJH,des}$ from the desorption branch of the isotherm and the diameter $D_{BJH,ads}$ from the adsorption branch by localizing the D_p axis position of the peak maximum of the calculated pore size distributions $\Delta V_{p,des}/\Delta D_p$ and $\Delta V_{p,ads}/\Delta D_p$ (1st linear derivatives of the cumulative pore volume vs. pore diameter), respectively.

Transport and storage

The material should be stored in the closed bottle at ambient temperature (20 to 25 °C) in a dry atmosphere.

Metrological traceability

The certified values are method-defined (model dependent) parameters. They were determined on the basis of the BET model and the BJH model as described in ISO 9277 and ISO 15901-2, respectively. Under the condition that these models are applied as an integral part of the traceability statement, the certified values are traceable to the base units of the SI via calibrated measurements of the quantities pressure, volume, and mass.

Participating laboratories

Aqura GmbH, Hanau (Germany)
BAM Federal Institute for Materials Research and Testing, Div. 1.3, Berlin (Germany)
BAM Federal Institute for Materials Research and Testing, Div. 5.6, Berlin (Germany)
Bayer Technology Services GmbH, Leverkusen (Germany)
Bayerisches Zentrum für Angewandte Energieforschung e.V., Würzburg (Germany)
BEL Japan Inc., Osaka (Japan)
Delft Solids Solutions, Delft (The Netherlands)
Fraunhofer-Institut für Keramische Technologien und Systeme, Hermsdorf (Germany)
Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Berlin (Germany)
Institute of Chemical and Engineering Sciences Ltd., Jurong Island (Singapore)
Instituto Pedro Nunes, Coimbra (Portugal)
Micromeritcs Instrument Corp., Norcross, GA (USA)
Micromeritics GmbH, Mönchengladbach (Germany)
POROTEC GmbH, Hofheim (Germany)
Quantachrome GmbH, Odelzhausen (Germany)
Quantachrome Instruments, Boynton Beach (USA)
Rubotherm - Präzisionsmesstechnik GmbH (Germany)
Sachtleben Chemie GmbH (Germany)
The Ural Research Institute for Metrology, Yekaterinburg (Russian Federation)
ThermoFisher Scientific, Milan (Italy)
University of Alicante (Spain)

Data set means of the participants in the interlaboratory certification study

Data set no.	A_{BET} (m^2/g)	$V_{p,0.99}$ (cm^3/g)	$4V_p/A_{BET}$ (nm)	$D_{BJH(des)}$ (nm)	$D_{BJH(ads)}$ (nm)
01	99.28	0.2402	9.68	8.08	11.10
02	95.41	0.2331	9.77	8.22	11.29
03	96.95	0.2328	9.60	7.77	12.27
04	95.91	0.2307	9.62	7.77	12.27
05	101.11	0.2398	9.49	8.13	11.25
06	96.75	0.2374	9.82	8.34	11.16
07	94.97	0.2309	9.73	8.27	11.65
08	95.90	0.2296	9.57	8.19	11.62
09	103.44 [a]	0.2456 [a]	9.50	8.14	11.31
10	97.06	0.2334	9.62	8.26	12.10
11	94.96	0.2336	9.84	8.36	10.70
12	96.19	0.2323	9.66	8.44	12.21
13	96.34	0.2335	9.70	8.00	11.11
14	95.65	0.2325	9.72	8.65	12.04
15	96.38	0.2329	9.66	8.22	12.44
16	100.87	0.2395	9.54	7.86	11.21
17	96.42	0.2360	9.80	8.20	12.20
18	97.73	0.2341	9.58	8.29	11.17
19	95.97	0.2308	9.63	8.19	11.32
20	97.13	0.2339	9.63	7.41 [a]	10.66
21	97.08	0.2360	9.72	8.40	11.26
22	96.23	0.2402	9.98	7.78	10.74
23	90.81	0.2274	10.02	6.00 [a]	9.22 [a]

[a] data set mean for the particular property statistically detected as outlier

References

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Certification Report of BAM-P106
BAM Federal Institute for Materials Research and Testing, Berlin 2012
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Guidelines for the Production of BAM Reference Materials.
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www.bam.de/en/fachthemen/referenzmaterialien

ISO Guide 35
Certification of reference materials - General and statistical principles.
ISO, Geneva 2006

ISO 9277
Determination of the specific surface area of solids by gas adsorption – BET method.
ISO, Geneva 2010

S. J. Gregg, K. S. W. Sing
Adsorption, Surface Area and Porosity.
Academic Press, London 1982

ISO 15901-2
Pore size distribution and porosity of solid materials by mercury porosimetry and gas adsorption – Part 2: Analysis of mesopores and macropores by gas adsorption
ISO, Geneva 2006, amd. 2007

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BAM Federal Institute for Materials Research and Testing

Prof. Dr. U. Panne
Head of Department 1
Analytical Chemistry;
Reference Materials

BUNDESANSTALT FÜR MATERIALFORSCHUNG UND -PRÜFUNG (BAM) 50

Dr. F. Emmerling
Head of Division 1.3
Structure Analysis

This reference material is supplied by:

BAM Federal Institute for Materials Research and Testing

Richard-Willstaetter-Str. 11, D-12489 Berlin, Germany

Tel: +49 30 8104 2061
Fax: +49 30 8104 1117

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