Certified Reference Material

BAM-P105

ausverkauft / out of stock

Pore size parameters of nanoporous glass

calculated from the nitrogen isotherm measured at 77.3 K

Certified Values

Property [a]		Value	Uncertainty U [b]	Unit
BET Specific Surface Area	A_{BET} [c]	197.0	4.0	m^2/g
Specific pore volume	V_p	0.2327	0.0025	cm^3/g
Hydraulic pore diameter	D_1 [d]	4.73	0.10	nm
Most frequent pore diameter	D_2 [e]	4.38	0.14	nm
Most frequent pore diameter	D_3 [f]	5.80	0.27	nm

[a] Certified in accordance with the Guidelines for the Production of BAM Reference Materials [1].

[b] The uncertainty was calculated as $U = k \cdot u_c$ where u_c is the combined standard uncertainty for the average with the coverage factor k = 2. The value of u_c includes both a combined estimate of the variation of the averages and the variation due to material inhomogeneity according to ISO Guide 35 [2].

[c] Recertified Value of BET Specific Surface Area in May 2016 in accordance with Guidelines for the Development and Production of BAM Reference Materials [3] and BAM reference procedure 402 [4].

[d] Recertified Value of hydraulic pore diameter $D_1 = 4\,V_p/A_{BET}$

[e] $D_2 = D_{BJH}$(Des.) is the pore size at maximum of the pore size distribution $\Delta V_p/\Delta D_p$ calculated from the desorption branch of the isotherm by applying the BJH model [5].

[f] $D_3 = D_{BJH}$(Ads.) is the pore size at maximum of the pore size distribution dV_p/dD_p calculated from the adsorption branch of the isotherm by applying the BJH model [5].

End of Validity

BAM-P105 did not prove to be as stable as originally assumed and a recertification was performed. This certificate replaces the original certificate of February 5, 2009 and is valid until January 2019.

Material Description

A unit of BAM-P105 consists of a single glass bottle containing approximately 10 g of a nanoporous glass material.

Recommended Use

The reference material is intended for the calibration and checking of instruments used for the determination of the BET specific surface area, the specific pore volume, the hydraulic pore diameter and the 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. Starting at room temperature, the sample is to be heated up to 473 K (200 °C) in vacuum within 1 hour followed by degassing the sample at 200 °C for 3 hours. 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. Choose sample size and perform analysis following the instrument manufacturer's instructions.

Determine the BET specific surface area in accordance with the international standard ISO 9277 [6] 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 nm^2 for the cross sectional area of nitrogen [6], [7].

The Single Point Total Pore Volume V_p according to the Gurvich rule should be determined from the adsorption branch of the isotherm at relative pressure $p/p_0 = 0.990$. Use the factor 0.001547 for density conversion of gaseous nitrogen at STP to liquid nitrogen at 77.3 K, i. e. $V_p(0.990) = 0.001547 \times V_a(0.990)$ [5], [7].

The relation $D_1 = 4V_p/A_{BET}$ has to be used for the calculation of the hydraulic pore diameter [5].

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 most frequent pore diameter D_2 from the desorption branch of the isotherm and the diameter D_3 from the adsorption branch by localizing the position of the peak maximum of the calculated pore size distributions $\Delta V_p(\text{ads.})/\Delta D_p$ and $\Delta V_p(\text{des.})/\Delta D_p$ (linear derivatives) respectively [5].

Transport and Storage

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

Participating Laboratories

Adsor-Tech GmbH, Premnitz (Germany)
AQura GmbH, Hanau (Germany)
BAM, Fachgruppe I.3, Berlin (Germany)
BAM, Fachgruppe V.4, 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 B.V., Delft (The Netherlands)
Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Berlin (Germany)
Hermsdorfer Institut für technische Keramik e. V. (HITK), Hermsdorf (Germany)
Micromeritics 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, Bochum (Germany)
Sachtleben Chemie GmbH, Duisburg (Germany)
ThermoFisher Scientific, Milan (Italy)
University of Alicante (Spain)
University of Amsterdam (The Netherlands)
University of Provence, Marseille (France)

Three of the laboratories participated in the interlaboratory study with two gas adsorption instruments each.

Certification Report

An accompanying report in German language [8] presenting the certification procedure and the statistical analyses as well as an amendment to this report describing the recertification procedure is available from BAM on request.

Literature

[1] Guidelines for the Production of BAM Reference Materials.
Bundesanstalt für Materialforschung und –prüfung (BAM), Berlin 2006

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

[3] Guidelines for the Development and Production of BAM Reference Materials.
Bundesanstalt für Materialforschung und –prüfung (BAM), Berlin 2016

[4] BAM Reference procedure 402 "Precision Measurement of the Specific Surface Area of Solids by Gas Adsorption
Bundesanstalt für Materialforschung und –prüfung (BAM), Berlin 2014

[5] 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

[6] ISO 9277
Determination of the specific surface area of solids by gas adsorption – BET method.
International Organization for Standardization, Geneva (2010)

[7] Sing, K. S. W., Everett, D. H., Haul, R. A. W., Moscou, L, Pierotti, R. A., Rouquerol, J. and Siemieniewska, T.
Reporting physical adsorption data for gas/solid systems with special reference to the determination of surface area and porosity. (IUPAC Recommendations 1984)
Pure Appl. Chem., 57 (1985) 603 - 619

[8] Klobes, P. Bremser, W., Zimathies, A. and Prinz, C.
Bericht zur Zertifizierung des Referenzmaterials BAM-P105
Bundesanstalt für Materialforschung und –prüfung (BAM), Berlin 2009

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