Certified Reference Material

BAM-P128

Porosity Properties of Macroporous Alumina Ceramic
Calculated from the Mercury Intrusion up to a Maximum Pressure
between 0.2 and 0.4 MPa

Certified Values

Property		Value [a]	Uncertainty U [b]	Unit
Specific pore volume [c]	V_p	220	6	mm^3/g
Median pore diameter [d]	d_{50}	27.6	1.0	µm

[a] Mean value of the means of accepted data sets each derived from at least 19 single values.

[b] Uncertainty $U = k \cdot u_c$ calculated according to ISO Guide 35 and ISO/IEC Guide 98 with the coverage factor k = 2 (giving a level of confidence of approximately 95 %). The combined standard uncertainty u_c of each certified property includes uncertainty contributions resulting from the interlaboratory testing, the study of inhomogeneities and stability of the material.

[c] Specific pore volume V_p calculated from the mercury intrusion with maximum pressure in a low-pressure device. Described in ISO 15901-1 and DIN 66133.

[d] Median pore diameter d_{50} calculated according to the Washburn equilibrium model as described in ISO 15901-1 and DIN 66133.

Informative Value

Property		Value	Uncertainty	Unit
Density	ρ_s	3.6405	0.0019	g/cm^3

This certificate is valid for 3 years after dispatch.

Date of dispatch: _____

Material Description

A unit of BAM-P128 consists of six cylinders of macroporous extruded and burnt alumina ceramic (Al_2O_3) material.

Recommended Use

The reference material is intended for performance testing of mercury porosimeters in the low-pressure device in the range up to 0.4 MPa for the determination of the specific pore volume and the median pore diameter.

Handling

The recommended sample intake is one cylinder per experiment.

Prior to the analysis, the sample material should be tempered at 105 °C in a drying oven for three hours.

Use mercury with a purity of 99.99 % or higher.

The transformation of the intrusion pressure data p_{Hg} into pore diameter values d_p according to the Washburn equation $d_p = -4\,\gamma\cos\theta / p_{Hg}$ (assuming a cylindrical pore model) has to be carried out using the following parameter values: γ = 0.48 N m^{-1} (surface tension of mercury) and θ = 140° (contact angle of the mercury) according to DIN 66133 and ISO 15901-1.

Transport and Storage

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

Participating Laboratories

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AQura GmbH, Marl (Germany)
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BAM Federal Institute for Materials Research and Testing, Div. 7.1, Berlin (Germany)
Forschungsinstitut für Anorganische Werkstoffe – Glas/Keramik- GmbH, Höhr-Grenzhausen (Germany)
Fraunhofer-Institut für Angewandte Polymerforschung IAP, Potsdam (Germany)
Fraunhofer-Institut für Solare Energiesysteme ISE, Freiburg (Germany)
Hoffmann & Co Elektrokohle AG, Bad Goisern (Austria)
Institut für Technische Chemie an der Universität Leipzig, Leipzig (Germany)
Leibniz-Institut für Oberflächenmodifizierung e.V., Leipzig (Germany)
Micromeritics GmbH, Aachen (Germany)
Micromeritics Instrument Corp., Norcross, GA (USA)
POROTEC GmbH, Hofheim (Germany)
Quantachrome GmbH, Odelzhausen (Germany)
Quantachrome Instruments, Boynton Beach (USA)
Rauschert Kloster Veilsdorf GmbH, Veilsdorf (Germany)
Technische Universität Dresden, Dresden (Germany)
Technische Universität München, München (Germany)
ThermoFisher Scientific, Milan (Italy)

One laboratory participated in the interlaboratory study with two instruments.

Results of participants in the interlaboratory certification study

Property $x \rightarrow$	V_p	d_{50}
Data set no. ↓	mm³/g	µm
01	---[a]	26.76
02	224.84	26.23
03	213.41	28.34
04	220.58	29.51
05	217.44	26.28
06	216.96	26.95
07	217.19	28.09
08	223.08	26.08
09	217.83	28.93
10	212.00	29.98
11	223.36	26.22
12	224.50	28.95
13	215.23	28.28
14	222.86	26.92
15	209.48	28.34
16	237.44	26.09
17	223.44	27.15
18	212.08	26.33
19	225.52	29.67
20	220.20	26.91
l	19	20
$\bar{x}$ [b]	219.865	27.600
s_x [c]	6.459	1.319
$\bar{x} + 1 \cdot s_x$	226.324	28.919
$\bar{x} - 1 \cdot s_x$	213.406	26.281
$\bar{x} + 2 \cdot s_x$	232.783	30.238
$\bar{x} - 2 \cdot s_x$	206.947	24.962

[a] value excluded for technical reasons.

[b] average of the accepted data set means for the particular property on the basis of 5 single measurements each.

[c] standard deviation of the data set for the particular property.

Metrological Traceability

The certified values are method-defined (model dependent) parameters. They were determined on the basis of the pore volume and median pore diameter as described in DIN 66133 and ISO 15901-1, 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.

Literature

Guidelines for the Production of BAM Reference Materials
Version 1, 20 June 2006, updated April 2010
BAM Bundesanstalt für Materialforschung und -prüfung, Berlin 2010
http://www.bam.de/en/fachthemen/referenzmaterialien/referenzmaterialien_medien/bam_rm_guidelines.pdf

ISO Guide 35
Certification of reference materials - General and statistical principles.
International Organization for Standardization, Geneva (2006)

ISO 15901-1
Evaluation of pore size distribution and porosimetry of solid materials by mercury porosimetry and gas adsorption– Part 1: Mercury porosimetry
International Organization for Standardization, Geneva (2007)

DIN 66133
Bestimmung der Porenvolumenverteilung und der spezifischen Oberfläche von Feststoffen durch Quecksilberintrusion.
DIN, Berlin (1993)

Emmerling, F., Bremser, W., Zimathies, A., and Prinz, C.
Certification Report for the Certified Reference Material BAM-P128
BAM –Federal Institute for Materials Research and Testing, Berlin 2015
Web-Link: http://www.rm-certificates.bam.de/en/certificates/porous_materials/index.htm

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