Federal Institute for Materials Research and Testing

CERTIFIED REFERENCE MATERIAL FOR MERCURY INTRUSION

BAM-P125
Material: Flat Membrane

Certificate

Certified properties

Mercury intrusion curve between 0.12 MPa and 0.88 MPa

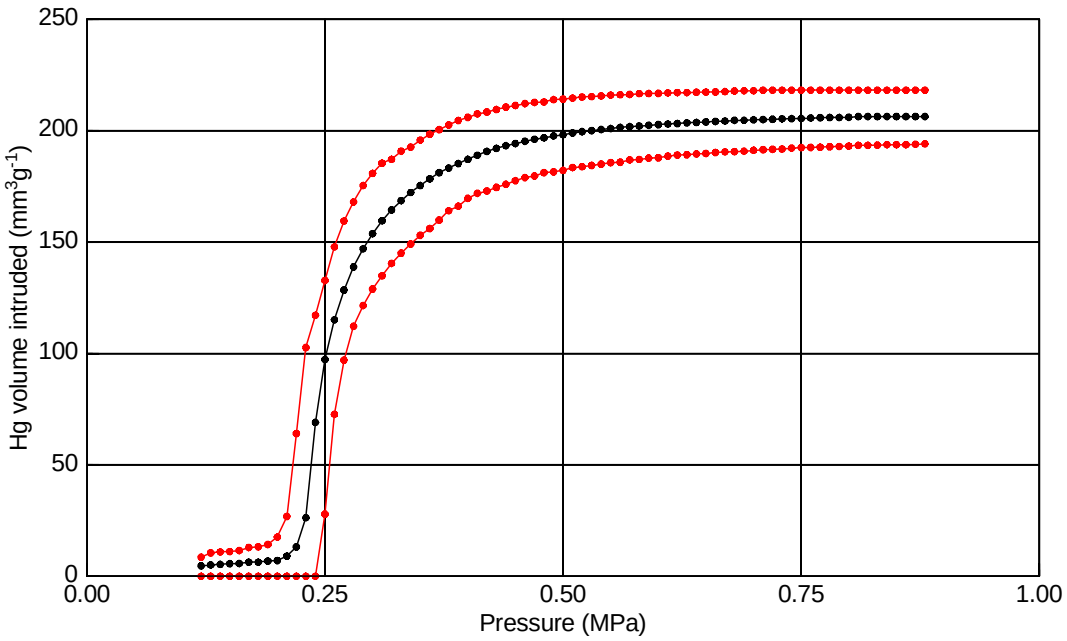

Figure 1: *Reference curve (black) with simultaneous prediction band at the significance level 0.95 (red) of the material BAM-P125 (for discrete values see annex)*

Mercury intrusion curve characteristics

Quantity	Certified value [1]	Uncertainty U [2]	Unit
y_1 [3]	207.9	197.8 - 218.0	mm^3g^{-1}
y_2 [4]	0.2646	0.2511 - 0.2782	MPa
y_3 [5]	0.1366	0.1187 - 0.1546	MPa
p_{50}	0.2554	0.2460 - 0.2649	MPa
d_{50}	5.797	5.581 - 6.012	µm

[1] Mercury intrusion curves from the designed interlaboratory testing were analysed by means of a multivariate variance components model for the curve characteristics y_1, y_2 and y_3. The results were mean curve characteristics (certified values) and prediction intervals for the curve characteristics. Adjusted curves and statistics from the variance components model were used to create a certified pressure volume curve with a prediction band.

[2] The prediction interval ±U at the significance level 0.95 results from the variance analytical investigation of the pressure volume curve characteristics y_1, y_2, and y_3 (see Figure 2).

[3] y_1: Intruded volume at the saturation point 0.88 MPa (saturation value).

[4] y_2: Pressure at 57.5 % of the saturation value. This value has been determined by local polynomial estimation (Epanechnikov kernel with band width h = 0.025 MPa).

[5] y_3: Difference of the pressures at which the intrusion curve has got 87.5 % and 25 % respectively of the saturation value (see Figure 2).

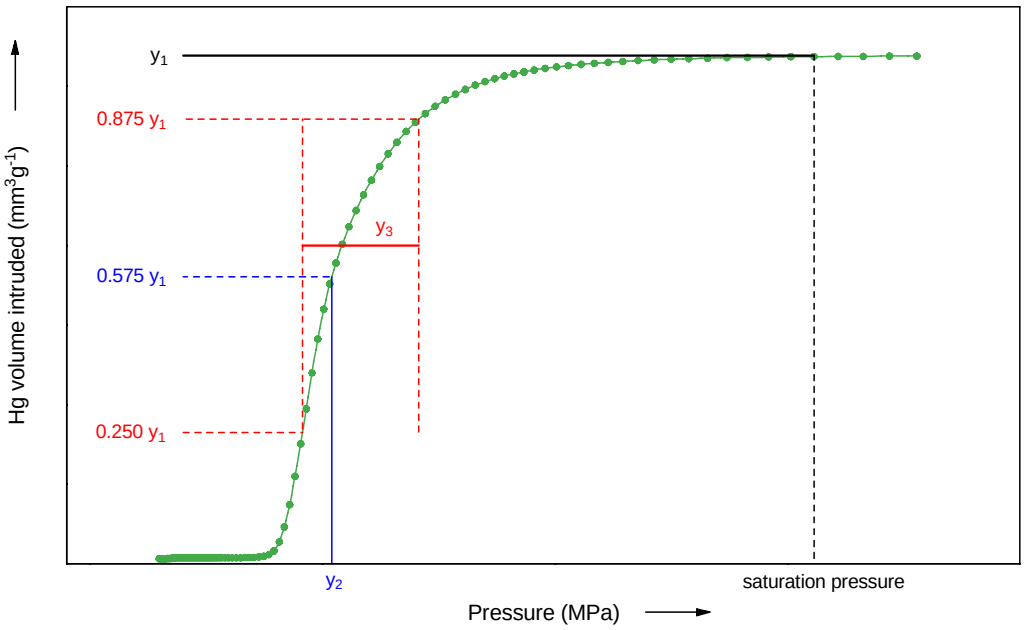

Figure 2: *Definition of the pressure-volume curve characteristics*

Non-certified properties*

Quantity	Value	Unit
Specific surface area	0.3	$m^2\ g^{-1}$
Bulk density	2.1	$g\ cm^{-3}$
Apparent density	3.9	$g\ cm^{-3}$
Porosity	45	%

* only as additional information, given without uncertainty

DESCRIPTION OF THE SAMPLE

The reference material consists of flat membranes of α-alumina produced by the Institut für Technische Keramik, e.V. (HITK), Hermsdorf, Germany. The whole batch of the material was divided into 50 plates. Each plate of the sintered material was cut into 30 pieces, numbered by means of a laser beam. In contrast to dispersed materials, in the case of the compact samples there is no possibility of homogenizing the whole candidate material. Therefore, the homogeneity of the batch was tested inside the experimental design of the interlaboratory testing for certification.

INSTRUCTION FOR USE

The reference material is intended for checking the performance of mercury porosimeters in the low-pressure range between 0.12 and 0.88 MPa.
The closed bottle should be stored at ambient temperature in a dry place.
Prior to the analysis, a heating procedure for drying the sample is not necessary if the sample is handled as described.
The recommended sample intake is one piece flat membrane per experiment.
Use mercury with a purity of 99.99 % (outgassed) or better.

DATA EVALUATION

– Measure one piece of the membranes and plot your measured pressure volume curve in one diagram with the reference curve and the prediction band (see Figures 1, 3, and 4).

– If the volume and pressure sensors of the porosimeter are correctly calibrated the measured curve lies, with the specified probability, completely between the curves defining the upper and the lower limit of the prediction band at level (1–α).

– The prediction band is defined as follows: A prediction band at level (1–α) covers the measured curve over the given pressure interval (0.12 to 0.88 MPa) completely with the specified probability. The size of prediction bands depends on the number of measured points per curve. Bands given here require about 60 measured points per curve.

– 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: $\gamma = 0.48$ N m^{-1} (surface tension of mercury) and $\theta = 140°$ (contact angle of the mercury) according to DIN 66133.

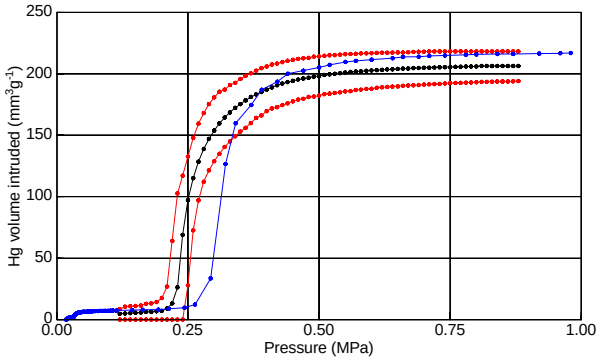

Figure 3: *Demonstration of a pressure sensor error of the porosimeter*

black - reference curve
red - prediction band at 0.95 significance level
blue- test curve

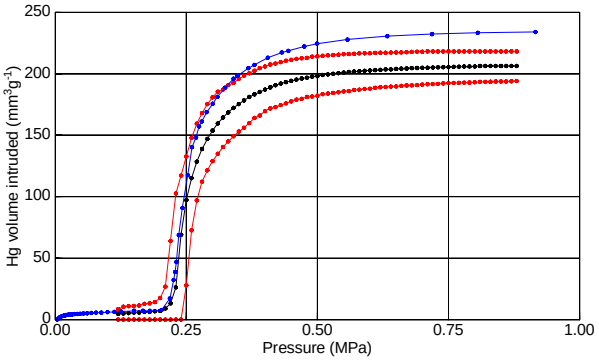

Figure 4: *Demonstration of a volume calibration error of of the porosimeter*

black - reference curve
red - prediction band at 0.95 significance level
blue- test curve

PARTICIPATING LABORATORIES

Co-ordination

BAM Bundesanstalt für Materialforschung und -prüfung, DE

Participants:

- BAM Bundesanstalt für Materialforschung und -prüfung, Berlin, DE
- Degussa AG, Hanau, DE
- DMT - Gesellschaft für Lehre und Bildung mbH, Bochum, DE
- Dr. C. Otto Feuerfest GmbH, Bochum, DE
- Eidgenössische Materialprüfungs- und Forschungsanstalt (EMPA), Dübendorf, CH
- Fraunhofer-Institut für Bauphysik, Valley, DE
- Hermsdorfer Institut für Technische Keramik e.V., Hermsdorf/Thür., DE
- Quantachrome GmbH, Odelzhausen, DE
- Technische Universität Dresden, Dresden, DE
- Technische Universität Hamburg-Harburg, Hamburg, DE
- ThermoQuest Italia S.p.A., CE Instruments, Rodano (Milan), IT
- Universität Gesamthochschule Kassel, DE

ANALYTICAL METHODS USED

Mercury intrusion according to DIN 66133

DOCUMENTATION

Guidelines for the production and certification of BAM reference materials

BCR/01/97 (1997)	Guidelines for the production and certification of BCR reference materials
ASTM D 4284-92	Standard test method for determining pore volume distribution of catalysts by mercury intrusion porosimetry
BS 7591-1 (1992)	Porosity and pore size distribution of materials Method of evaluation by mercury porosimetry
DIN 66133 (1993)	Bestimmung der Porenvolumenverteilung und der spezifischen Oberfläche von Feststoffen durch Quecksilberintrusion

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Overall co-ordination of this certification	:	B. Röhl-Kuhn
Statistics	:	Jörg Polzehl, Weierstraß-Institut Berlin

Certification date: 2003-12-02

This certificate is valid for three years after purchase.

Sales date:

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Head of Department
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Reference Materials

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Reference Materials

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Annex

Discrete values of the reference curve with simultaneous prediction bands

Data Point No.	p_{Hg} (MPa)	$V_{Hg} - U$ (mm^3g^{-1})	V_{Hg} (mm^3g^{-1})	$V_{Hg} + U$ (mm^3g^{-1})
1	0.12	0.000	4.634	8.533
2	0.13	0.000	5.032	10.477
3	0.14	0.000	5.301	10.922
4	0.15	0.000	5.603	11.044
5	0.16	0.000	5.695	11.492
6	0.17	0.000	6.293	12.856
7	0.18	0.000	6.293	13.202
8	0.19	0.000	6.789	14.211
9	0.20	0.000	7.029	17.532
10	0.21	0.000	9.050	26.819
11	0.22	0.000	13.195	64.042
12	0.23	0.000	26.246	102.680
13	0.24	0.000	69.024	117.130
14	0.25	27.858	97.281	132.770
15	0.26	72.641	115.130	147.890
16	0.27	97.025	128.500	159.490
17	0.28	112.220	138.850	168.060
18	0.29	121.520	147.050	175.420
19	0.30	128.960	153.820	180.870
20	0.31	134.910	159.650	185.480
21	0.32	140.460	164.510	187.180
22	0.33	145.040	168.650	190.870
23	0.34	149.150	172.280	192.690
24	0.35	153.060	175.420	195.810
25	0.36	156.100	178.430	198.450
26	0.37	159.940	181.200	200.550
27	0.38	164.090	183.300	202.610
28	0.39	166.140	185.300	204.630
29	0.40	169.660	187.190	206.040
30	0.41	171.930	189.030	207.590
31	0.42	172.950	190.740	208.420
32	0.43	174.560	192.100	209.540
33	0.44	175.950	193.310	210.650
34	0.45	177.590	194.270	211.370
35	0.46	179.050	195.310	212.220
36	0.47	179.730	196.220	212.700
37	0.48	181.220	196.860	212.980
38	0.49	181.570	197.730	213.960
39	0.50	182.260	198.230	214.210

Data Point No.	p_{Hg} (MPa)	$V_{Hg} - U$ (mm^3g^{-1})	V_{Hg} (mm^3g^{-1})	$V_{Hg} + U$ (mm^3g^{-1})
40	0.51	183.490	199.020	214.670
41	0.52	183.910	199.600	215.150
42	0.53	184.480	200.070	215.320
43	0.54	185.050	200.570	215.630
44	0.55	185.700	201.030	216.020
45	0.56	185.900	201.550	216.190
46	0.57	186.920	201.870	216.290
47	0.58	187.150	202.110	216.690
48	0.59	187.760	202.490	216.740
49	0.60	187.890	202.770	216.780
50	0.61	188.590	203.050	217.020
51	0.62	189.110	203.240	217.170
52	0.63	189.290	203.600	217.190
53	0.64	189.630	203.750	217.310
54	0.65	189.810	203.990	217.400
55	0.66	190.290	204.210	217.430
56	0.67	190.560	204.360	217.590
57	0.68	190.660	204.680	217.870
58	0.69	190.840	204.750	217.990
59	0.70	191.250	204.960	218.000
60	0.71	191.540	205.020	218.240
61	0.72	191.730	205.170	218.240
62	0.73	191.830	205.390	218.240
63	0.74	192.240	205.470	218.240
64	0.75	192.490	205.510	218.240
65	0.76	192.550	205.710	218.240
66	0.77	192.660	205.850	218.240
67	0.78	192.860	205.970	218.240
68	0.79	192.960	205.970	218.240
69	0.80	193.170	206.070	218.240
70	0.81	193.500	206.410	218.240
71	0.82	193.510	206.410	218.240
72	0.83	193.570	206.410	218.240
73	0.84	193.770	206.410	218.240
74	0.85	193.770	206.410	218.240
75	0.86	193.770	206.410	218.240
76	0.87	193.980	206.410	218.240
77	0.88	194.160	206.410	218.240

V_{Hg} Certified pressure-volume curve (reference curve)

$V_{Hg} - U$ Lower limit curve of prediction band at significance level 0.95

$V_{Hg} + U$ Upper limit curve of prediction band at significance level 0.95