Federal Institute for Materials Research and Testing

CERTIFIED REFERENCE MATERIAL FOR MERCURY INTRUSION

BAM-P124
Material: Flat Membrane

Certificate

Certified properties

Mercury intrusion curve between 0.24 MPa and 1.55 MPa

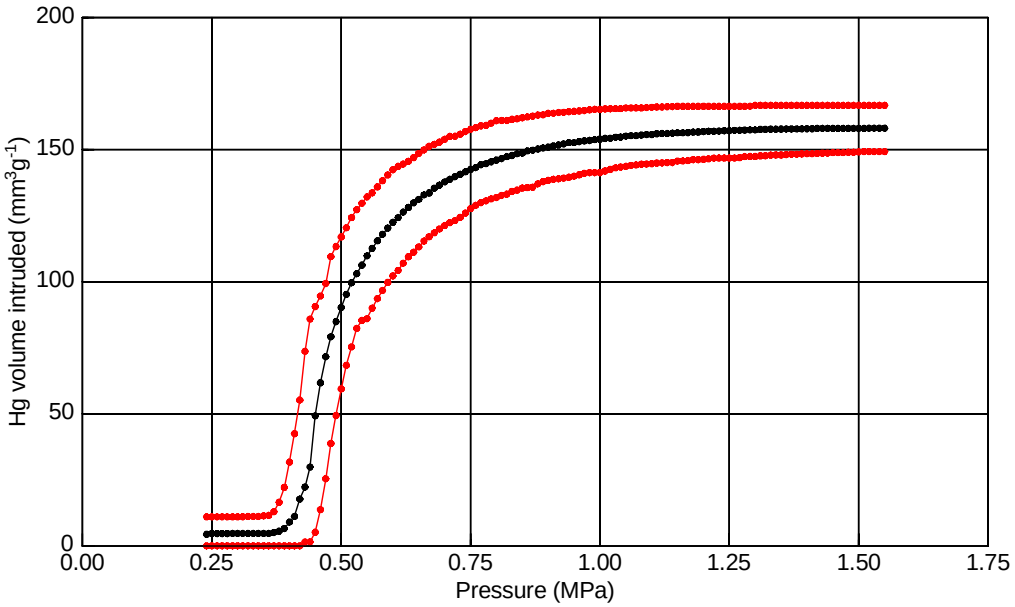

Figure 1: *Reference curve (black) with simultaneous prediction band at the significance level 0.95 (red) of the material BAM-P124 (for discrete values see annex)*

Mercury intrusion curve characteristics

Quantity	Certified value [1)]	Uncertainty U [2)]	Unit
y_1 [3)]	158.1	150.8 - 165.4	mm^3g^{-1}
y_2 [4)]	0.5021	0.474 - 0.530	MPa
y_3 [5)]	0.2616	0.223 - 0.300	MPa
p_{50}	0.4795	0.451 - 0.508	MPa
d_{50}	3.074	2.89 - 3.26	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 1.55 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).

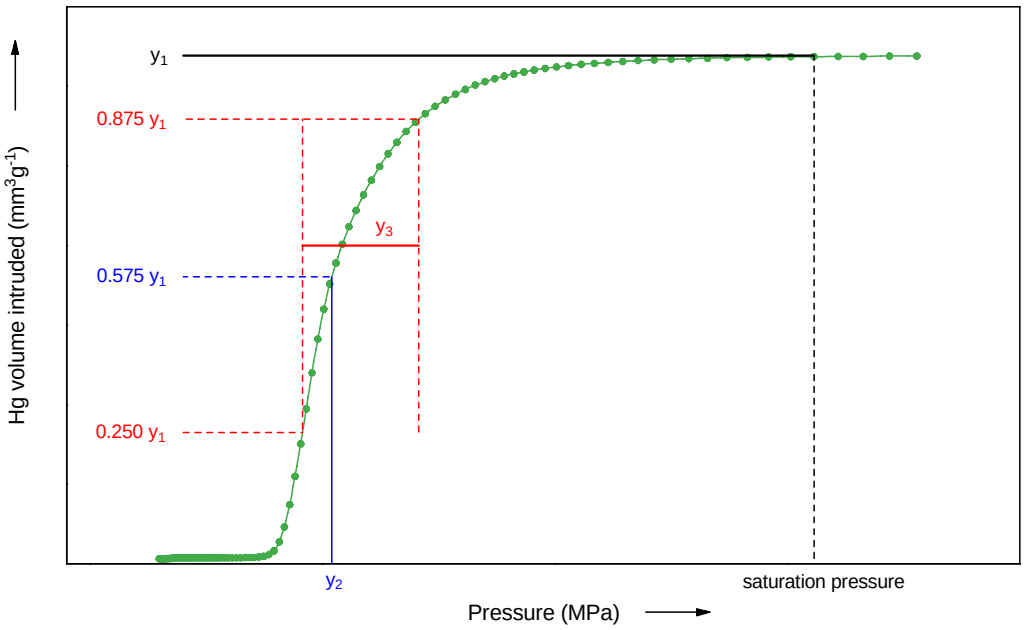

Figure 2: *Definition of the pressure-volume curve characteristics*

Non-certified properties*

Quantity	Value	Unit
Specific surface area	0.3	$m^2\ g^{-1}$
Bulk density	2.4	$g\ cm^{-3}$
Apparent density	3.9	$g\ cm^{-3}$
Porosity	38	%

* 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 0.24 and 1.55 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 specific 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 (0.24 to 1.55 MPa) completely with the specified probability. The size of prediction bands depends on the number of measured points per curve. Bands given here are for 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.

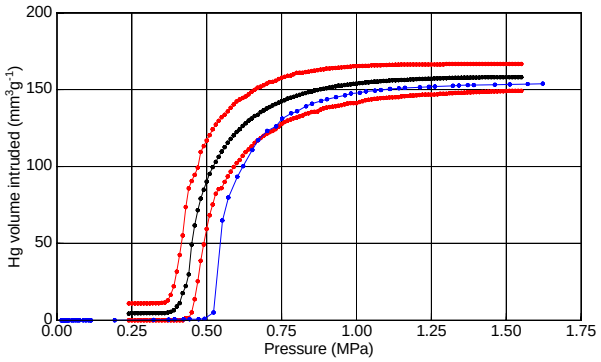

Figure 3 *Demonstration of a pressure sensor error of the porosimeter*

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

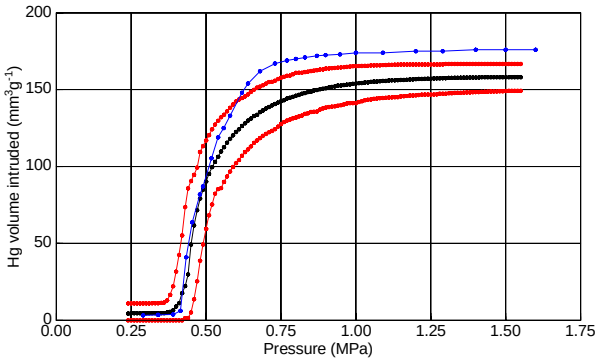

Figure 4 *Demonstration of a volume calibration error of of the porosimeter*

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
- Delft University of Technology, Delft, NL
- DMT - Gesellschaft für Lehre und Bildung mbH, Bochum, DE
- Eidgenössische Materialprüfungs- und Forschungsanstalt (EMPA), Dübendorf, CH
- Forschungsinstitut für anorganische Werkstoffe - Glas/Keramik - GmbH, Höhr-Grenzhausen, DE
- Fraunhofer-Institut für Bauphysik, Valley, DE
- Hermsdorfer Institut für Technische Keramik e.V., Hermsdorf/Thür., DE
- Micromeritics GmbH, Mönchengladbach, 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 Hannover, Hannover, 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

LEGAL NOTICE

BAM-project "Porous Reference Materials"	:	P. Klobes
Overall co-ordination of this certification	:	B. Röhl-Kuhn
Statistics	:	Jörg Polzehl, Weierstraß-Institut Berlin

Certification date: 2005-02-11

This certificate is valid for three years after purchase.

Sales date:

Professor Dr U Panne

Head of Department
Analytical Chemistry;
Reference Materials

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This reference material is provided by:

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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	0.24	0.000	4.333	10.982
2	0.25	0.000	4.636	10.982
3	0.26	0.000	4.636	10.982
4	0.27	0.000	4.636	10.982
5	0.28	0.000	4.636	10.982
6	0.29	0.000	4.677	10.982
7	0.30	0.000	4.677	10.982
8	0.31	0.000	4.677	11.004
9	0.32	0.000	4.677	11.107
10	0.33	0.000	4.677	11.107
11	0.34	0.000	4.677	11.107
12	0.35	0.000	4.677	11.336
13	0.36	0.000	4.683	11.535
14	0.37	0.000	5.070	12.924
15	0.38	0.000	5.545	16.519
16	0.39	0.000	6.576	22.105
17	0.40	0.000	9.015	31.699
18	0.41	0.000	11.146	42.449
19	0.42	0.000	17.697	55.188
20	0.43	1.468	22.277	73.652
21	0.44	1.521	29.853	85.863
22	0.45	5.155	49.238	90.580
23	0.46	13.708	61.706	94.610
24	0.47	25.406	71.594	99.300
25	0.48	38.744	79.179	109.500
26	0.49	49.326	84.881	113.320
27	0.50	59.438	90.224	116.930
28	0.51	68.346	95.211	120.440
29	0.52	75.312	99.631	124.250
30	0.53	82.346	103.020	127.280
31	0.54	85.336	106.290	129.700
32	0.55	86.042	109.770	132.100
33	0.56	90.002	112.640	133.660
34	0.57	93.621	115.480	135.920
35	0.58	96.681	117.950	138.240
36	0.59	99.732	120.320	140.390
37	0.60	102.220	122.490	142.410
38	0.61	104.280	124.260	143.650
39	0.62	107.010	126.370	144.570
40	0.63	109.430	128.110	145.640

Data Point No.	p_{Hg} (MPa)	$V_{Hg} - U$ (mm^3g^{-1})	V_{Hg} (mm^3g^{-1})	$V_{Hg} + U$ (mm^3g^{-1})
41	0.64	111.170	129.860	146.980
42	0.65	113.180	131.190	148.580
43	0.66	115.410	132.880	149.920
44	0.67	117.010	133.590	151.300
45	0.68	118.550	135.430	152.030
46	0.69	119.970	136.540	152.870
47	0.70	121.260	137.780	153.920
48	0.71	122.340	138.730	155.000
49	0.72	123.150	139.790	155.050
50	0.73	124.340	140.700	155.840
51	0.74	125.970	141.610	156.880
52	0.75	127.680	142.500	157.710
53	0.76	128.920	143.200	158.340
54	0.77	129.920	144.280	159.020
55	0.78	130.640	144.710	159.170
56	0.79	131.380	145.380	160.050
57	0.80	131.860	146.070	160.950
58	0.81	132.680	146.620	161.050
59	0.82	133.170	147.350	161.070
60	0.83	134.200	147.800	161.490
61	0.84	134.700	148.470	161.680
62	0.85	135.500	148.690	162.090
63	0.86	135.630	149.520	162.370
64	0.87	135.770	149.790	162.720
65	0.88	137.060	150.270	163.110
66	0.89	137.890	150.620	163.240
67	0.90	138.350	150.930	163.760
68	0.91	138.770	151.390	163.800
69	0.92	138.960	151.760	164.050
70	0.93	139.240	152.160	164.130
71	0.94	139.560	152.670	164.330
72	0.95	139.930	152.670	164.480
73	0.96	140.470	153.040	164.640
74	0.97	141.030	153.310	164.800
75	0.98	141.280	153.500	165.060
76	0.99	141.280	153.780	165.290
77	1.00	141.280	153.960	165.290
78	1.01	141.950	154.260	165.440
79	1.02	142.490	154.320	165.480
80	1.03	143.140	154.720	165.510

Annex

Discrete values of the reference curve with simultaneous prediction bands (cont.)

Data Point No.	p_{Hg} (MPa)	$V_{Hg} - U$ (mm^3g^{-1})	V_{Hg} (mm^3g^{-1})	$V_{Hg} + U$ (mm^3g^{-1})
81	1.04	143.440	154.780	165.520
82	1.05	143.690	155.080	165.800
83	1.06	144.050	155.250	165.800
84	1.07	144.210	155.250	165.820
85	1.08	144.520	155.480	165.830
86	1.09	144.560	155.700	165.960
87	1.10	144.690	155.700	166.030
88	1.11	144.900	156.000	166.130
89	1.12	145.010	156.120	166.130
90	1.13	145.120	156.120	166.260
91	1.14	145.130	156.220	166.300
92	1.15	145.670	156.380	166.400
93	1.16	145.740	156.380	166.400
94	1.17	145.880	156.440	166.400
95	1.18	146.240	156.650	166.400
96	1.19	146.240	156.700	166.400
97	1.20	146.310	156.870	166.400
98	1.21	146.540	156.960	166.400
99	1.22	146.760	156.960	166.400
100	1.23	146.800	157.040	166.400
101	1.24	146.820	157.150	166.400
102	1.25	146.820	157.150	166.400
103	1.26	146.820	157.320	166.400
104	1.27	146.970	157.350	166.400
105	1.28	147.360	157.400	166.400
106	1.29	147.390	157.490	166.450

Data Point No.	p_{Hg} (MPa)	$V_{Hg} - U$ (mm^3g^{-1})	V_{Hg} (mm^3g^{-1})	$V_{Hg} + U$ (mm^3g^{-1})
107	1.30	147.400	157.490	166.760
108	1.31	147.660	157.650	166.760
109	1.32	147.700	157.650	166.760
110	1.33	147.910	157.770	166.760
111	1.34	147.960	157.770	166.760
112	1.35	147.960	157.810	166.760
113	1.36	148.110	157.820	166.760
114	1.37	148.250	157.820	166.760
115	1.38	148.250	157.860	166.760
116	1.39	148.370	157.880	166.760
117	1.40	148.470	157.920	166.760
118	1.41	148.470	157.920	166.760
119	1.42	148.560	158.060	166.760
120	1.43	148.670	158.060	166.760
121	1.44	148.690	158.060	166.760
122	1.45	148.860	158.060	166.760
123	1.46	148.860	158.060	166.760
124	1.47	148.870	158.060	166.760
125	1.48	149.010	158.060	166.760
126	1.49	149.010	158.060	166.760
127	1.50	149.140	158.060	166.760
128	1.51	149.270	158.070	166.760
129	1.52	149.270	158.080	166.760
130	1.53	149.270	158.110	166.760
131	1.54	149.270	158.110	166.760
132	1.55	149.270	158.110	166.760

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