Federal Institute for Materials Research and Testing

CERTIFIED REFERENCE MATERIAL FOR MERCURY INTRUSION

BAM-P126
Material: Flat Membrane

Certificate

Certified properties

Mercury intrusion curve between 0.55 MPa and 2.1 MPa

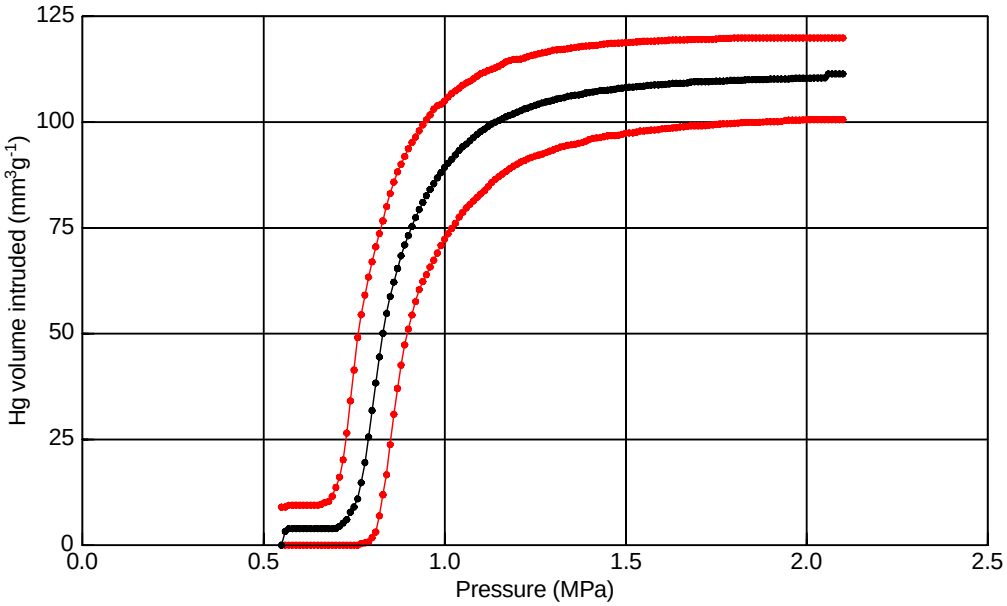

Figure 1: *Reference curve (black) with simultaneous prediction band at the significance level 0.95 (red) of the material BAM-P126 (for discrete values see annex)*

Mercury intrusion curve characteristics

Quantity	Certified value [1)]	Uncertainty U [2)]	Unit
y_1 [3)]	110.9	102.4 - 119.4	mm^3g^{-1}
y_2 [4)]	0.8682	0.8274 - 0.9091	MPa
y_3 [5)]	0.2965	0.2660 - 0.3271	MPa
p_{50}	0.8441	0.8025 - 0.8856	MPa
d_{50}	1.746	1.661 - 1.832	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 2.1 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 = 0.05 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).

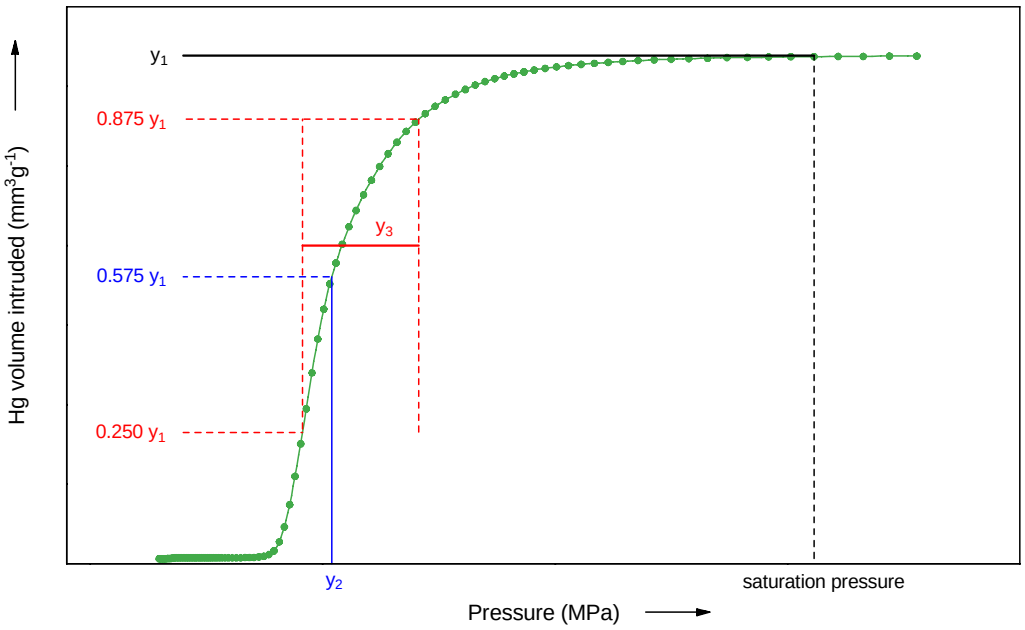

Figure 2: *Definition of the pressure-volume curve characteristics*

Non-certified properties*

Quantity	Value	Unit
Specific surface area	0.3	$m^2\ g^{-1}$
Bulk density	2.6	$g\ cm^{-3}$
Apparent density	3.8	$g\ cm^{-3}$
Porosity	30	%

* 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 0.55 and 2.1 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 (0.55 – 2.1 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.

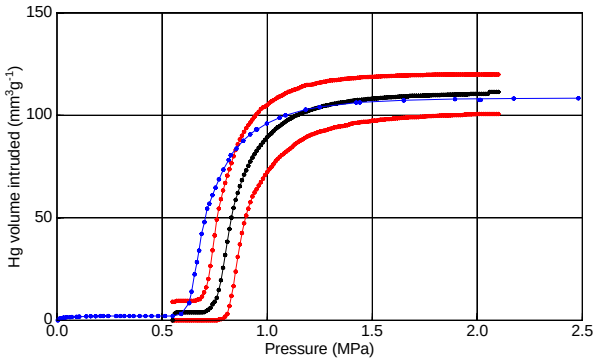

Figure 3: *Demonstration of a pressure sensor error of the porosimeter*

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

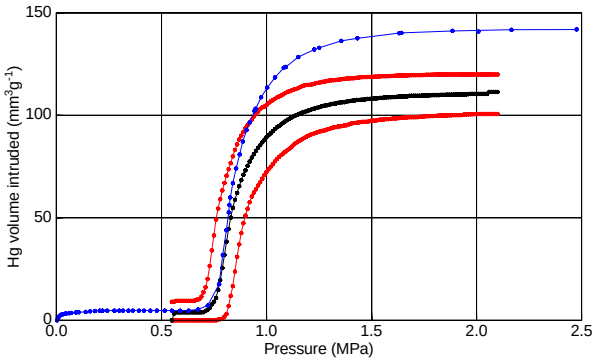

Figure 4: *Demonstration of a volume calibration error of of the porosimeter*

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
- Eidgenössische Materialprüfungs- und Forschungsanstalt (EMPA), Dübendorf, CH
- Forschungsinstitut für anorg. 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:

Prof Dr U Panne

Head of Department
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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	0.55	0.000	0.000	9.021
2	0.56	0.000	3.259	9.027
3	0.57	0.000	3.909	9.440
4	0.58	0.000	3.909	9.456
5	0.59	0.000	3.909	9.456
6	0.60	0.000	3.909	9.456
7	0.61	0.000	3.909	9.456
8	0.62	0.000	3.909	9.456
9	0.63	0.000	3.909	9.456
10	0.64	0.000	3.909	9.456
11	0.65	0.000	3.909	9.456
12	0.66	0.000	3.909	9.696
13	0.67	0.000	3.909	10.069
14	0.68	0.000	3.909	10.355
15	0.69	0.000	3.909	11.547
16	0.70	0.000	3.965	13.655
17	0.71	0.000	4.487	16.078
18	0.72	0.000	5.228	20.196
19	0.73	0.000	6.046	26.504
20	0.74	0.000	7.808	34.133
21	0.75	0.000	9.040	41.413
22	0.76	0.000	10.938	49.083
23	0.77	0.392	14.788	54.483
24	0.78	0.564	19.541	59.119
25	0.79	0.768	25.589	63.363
26	0.80	1.794	31.847	66.997
27	0.81	3.129	38.338	70.540
28	0.82	6.949	44.502	73.668
29	0.83	11.920	50.100	76.659
30	0.84	16.654	54.807	80.050
31	0.85	23.793	58.788	83.123
32	0.86	30.948	62.173	85.819
33	0.87	37.058	65.415	88.248
34	0.88	42.580	68.405	90.055
35	0.89	47.333	70.990	91.914
36	0.90	51.092	73.189	93.724
37	0.91	54.403	75.357	95.203
38	0.92	57.611	77.465	96.475
39	0.93	60.416	79.342	97.941
40	0.94	62.346	81.044	99.342

Data Point No.	p_{Hg} (MPa)	$V_{Hg} - U$ (mm^3g^{-1})	V_{Hg} (mm^3g^{-1})	$V_{Hg} + U$ (mm^3g^{-1})
41	0.95	63.927	82.624	100.560
42	0.96	65.766	84.101	101.720
43	0.97	67.352	85.449	103.070
44	0.98	69.057	86.821	103.880
45	0.99	70.841	88.046	104.240
46	1.00	72.309	89.316	104.980
47	1.01	73.622	90.310	105.930
48	1.02	74.872	91.240	106.800
49	1.03	76.114	92.265	107.390
50	1.04	77.550	93.304	108.020
51	1.05	78.658	94.172	108.730
52	1.06	79.740	94.824	109.260
53	1.07	80.585	95.576	109.670
54	1.08	81.378	96.365	110.260
55	1.09	82.254	97.124	110.900
56	1.10	83.040	97.816	111.420
57	1.11	83.781	98.350	111.760
58	1.12	84.704	98.964	112.200
59	1.13	85.731	99.563	112.540
60	1.14	86.484	99.997	112.830
61	1.15	87.150	100.370	113.230
62	1.16	87.770	100.830	113.720
63	1.17	88.336	101.280	114.270
64	1.18	89.009	101.650	114.580
65	1.19	89.569	101.970	114.820
66	1.20	90.056	102.350	114.860
67	1.21	90.546	102.670	114.920
68	1.22	90.896	103.110	115.170
69	1.23	91.335	103.340	115.460
70	1.24	91.679	103.670	115.750
71	1.25	92.007	103.970	115.950
72	1.26	92.240	104.270	116.170
73	1.27	92.443	104.550	116.360
74	1.28	92.778	104.780	116.530
75	1.29	93.120	104.940	116.770
76	1.30	93.408	105.190	117.020
77	1.31	93.782	105.490	117.090
78	1.32	94.117	105.640	117.180
79	1.33	94.291	105.760	117.240
80	1.34	94.536	106.100	117.340

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	1.35	94.652	106.240	117.530
82	1.36	94.747	106.350	117.630
83	1.37	95.015	106.410	117.770
84	1.38	95.155	106.620	117.920
85	1.39	95.503	106.890	117.980
86	1.40	95.912	107.020	118.040
87	1.41	96.174	107.130	118.110
88	1.42	96.293	107.290	118.150
89	1.43	96.380	107.480	118.310
90	1.44	96.573	107.480	118.440
91	1.45	96.752	107.580	118.540
92	1.46	96.819	107.660	118.640
93	1.47	96.819	107.770	118.640
94	1.48	96.872	107.990	118.680
95	1.49	97.130	108.010	118.710
96	1.50	97.383	108.170	118.760
97	1.51	97.442	108.260	118.820
98	1.52	97.443	108.260	118.890
99	1.53	97.666	108.390	118.970
100	1.54	97.907	108.450	119.020
101	1.55	97.914	108.480	119.120
102	1.56	97.999	108.660	119.150
103	1.57	98.134	108.750	119.150
104	1.58	98.135	108.810	119.160
105	1.59	98.217	108.810	119.230
106	1.60	98.413	108.900	119.250
107	1.61	98.461	108.920	119.270
108	1.62	98.525	109.030	119.390
109	1.63	98.641	109.170	119.420
110	1.64	98.701	109.170	119.420
111	1.65	98.837	109.170	119.420
112	1.66	98.890	109.180	119.430
113	1.67	99.003	109.270	119.480
114	1.68	99.122	109.480	119.540
115	1.69	99.130	109.550	119.580
116	1.70	99.130	109.550	119.580
117	1.71	99.130	109.550	119.580
118	1.72	99.155	109.550	119.580

Data Point No.	p_{Hg} (MPa)	$V_{Hg} - U$ (mm^3g^{-1})	V_{Hg} (mm^3g^{-1})	$V_{Hg} + U$ (mm^3g^{-1})
119	1.73	99.271	109.550	119.580
120	1.74	99.369	109.640	119.580
121	1.75	99.443	109.690	119.660
122	1.76	99.550	109.690	119.780
123	1.77	99.616	109.690	119.790
124	1.78	99.616	109.780	119.790
125	1.79	99.657	109.890	119.840
126	1.80	99.691	109.890	119.910
127	1.81	99.751	109.890	119.910
128	1.82	99.893	109.890	119.910
129	1.83	99.949	109.990	119.910
130	1.84	99.949	110.010	119.910
131	1.85	99.949	110.040	119.910
132	1.86	99.949	110.060	119.910
133	1.87	99.961	110.060	119.910
134	1.88	100.040	110.060	119.910
135	1.89	100.100	110.090	119.910
136	1.90	100.100	110.190	119.910
137	1.91	100.100	110.190	119.910
138	1.92	100.100	110.190	119.910
139	1.93	100.160	110.190	119.910
140	1.94	100.380	110.190	119.910
141	1.95	100.460	110.190	119.910
142	1.96	100.470	110.320	119.910
143	1.97	100.470	110.390	119.910
144	1.98	100.490	110.390	119.910
145	1.99	100.540	110.390	119.910
146	2.00	100.600	110.390	119.910
147	2.01	100.610	110.390	119.910
148	2.02	100.610	110.420	119.910
149	2.03	100.610	110.430	119.910
150	2.04	100.610	110.430	119.910
151	2.05	100.610	110.470	119.910
152	2.06	100.610	111.410	119.910
153	2.07	100.610	111.410	119.910
154	2.08	100.610	111.410	119.910
155	2.09	100.610	111.410	119.910
156	2.10	100.610	111.410	119.910

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