BAM

Federal Institute for Materials Research and Testing

CERTIFIED REFERENCE MATERIAL FOR MERCURY INTRUSION

BAM-P124
Material: Flat Membrane

Certificate

Certified properties:

Pressure-volume curve between 0,24 MPa and 1,55 MPa of mercury intrusion

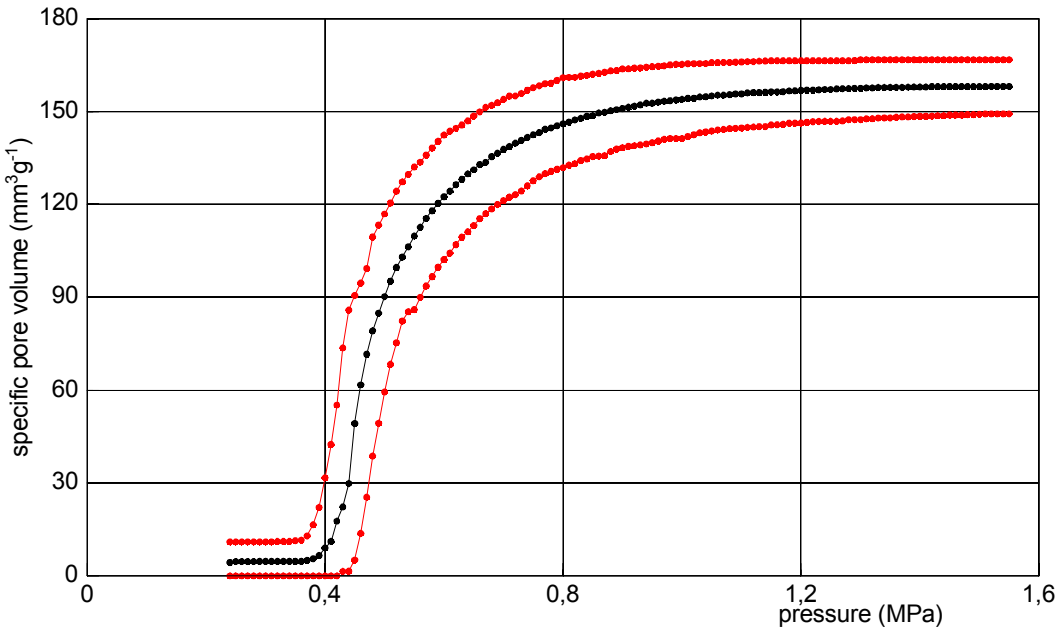

Figure 1 Reference curve (black) with simultaneous prediction band at the significance level 0,95 (red) for the material BAM-P124 (for discrete values see annex)

Certified discrete values of BAM-P124 (curve characteristics)

Quantity	Certified values	0,95- Confidence Interval	Unit
y_1 $V_{p,\ 1,55\ MPa}$ (specific pore volume at 1,55 MPa)	158,1	157,5 - 158,7	mm^3g^{-1}
y_2	0,5021	0,4999 - 0,5043	MPa
y_3	0,2616	0,2585 - 0,2647	MPa
p_{50}	0,4795	0,4772 - 0,4818	MPa
d_{50}	3,074	3,059 - 3,088	µm

Quantity	Certified values	0,95- Prediction Interval	Unit
y_1 $V_{p,\ 1,55\ MPa}$ (specific pore volume at 1,55 MPa)	158,1	150,8 - 165,4	mm^3g^{-1}
y_2	0,5021	0,474 - 0,530	MPa
y_3	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

0

The confidence intervals and the prediction intervals result from the variance analytical investigation of the p-v curve characteristics y_1 (intruded volume at the saturation point), y_2 (the $p_{57,5}$-value) and y_3 *(see below and Figure* 2). The determination of the curve characteristics are described in the certification report in detail.

y_1 : intruded volume at the saturation point 1,55 MPa (saturation value)
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)
y_3 : difference of the pressures at which the smoothed curve has got 87,5 % resp. 25 % of the saturation value (see Figure 2)

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 cylindric pore model) has to be carried out using the following values of the parameters: γ = 0.48 N m^{-1} (surface tension of mercury) and θ = 140° (contact angle of the mercury) according to DIN 66133.

The prediction interval is relevant for the user, especially for the specific pore volume y_1 resp. the pressure p_{50} and the diameter d_{50}.

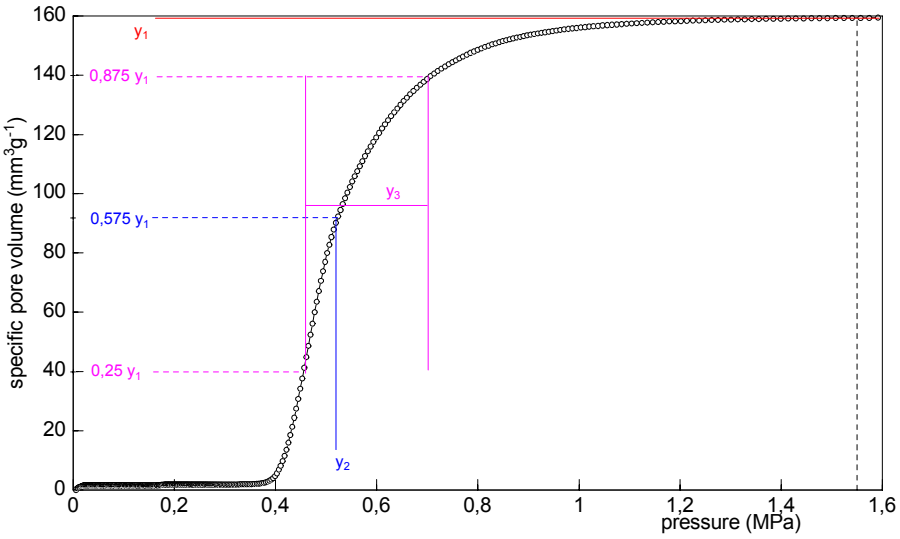

Figure 2 Standardization of the pressure-volume curves (see certification report)

Non-certified properties*:

Quantity	Unit	Value
Specific surface area	$m^2 g^{-1}$	0,3
Bulk density	$g\ cm^{-3}$	2,4
Apparent density	$g\ cm^{-3}$	3,9
Porosity	%	38

* only as additional information, given without uncertainty

DESCRIPTION OF THE SAMPLE

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

INSTRUCTION FOR USE

The reference material is intended for use in the calibration and especially for the checking of the low pressure range of mercury porosimeters in the range between 0,24 and 1,55 MPa.
The closed bottle should be stored at ambient temperature in a dry place.
The required sample intake is 1 piece flat membrane per experiment.
Use mercury with a purity of 99,99 % (outgassed) or better.
Prior to the analysis, a heating procedure for drying the sample is not necessary, if the sample is handled as described.

DATA EVALUATION

- Measure 1 piece of the membranes and put your measured pressure-volume curve into the diagramm with the reference curve and the prediction bands, see Figure 1.

- If the volume and pressure sensors of the porosimeter had been correctly calibrated the measured curve lies, with the selected probability, completely between the curves defining the bounds of the prediction band of level (1-α).

- Definition of the prediction band:
  A prediction band of level (1-α) covers the measured curve over the given pressure interval (0,24 – 1,55 MPa) completely with the selected 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.

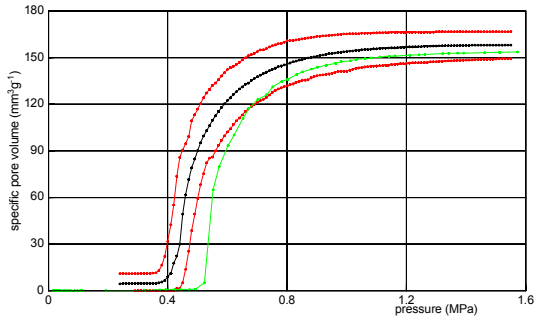

Figure 3 Demonstration of the calibration error in the pressure sensor of the device
reference curve - black
prediction band with 0,95 significance level - red
test curve - green

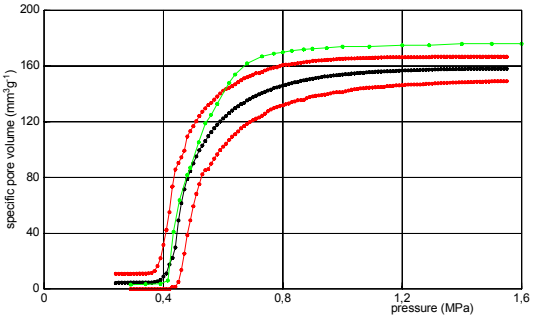

Figure 4 Demonstration of the calibration error of the volume of the device
reference curve - black
prediction band with 0,95 significance level - red
test curve - green

SHELF LIFE

Provided the sample is stored and handled appropriately, the certification will remain valid for 24 months from the date of shipment.

PARTICIPATING LABORATORIES

Co-ordination

Bundesanstalt für Materialforschung und -prüfung, DE

Participants:

- Bundesanstalt für Materialforschung und -prüfung (BAM), (5 equipments in 2 laboratories), 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), CH
- Forschungsinstitut für anorganische Werkstoffe - Glas/Keramik - GmbH, Höhr-Grenzhausen, DE
- Fraunhofer-Institut für Bauphysik, Valley-Oberlaindern, DE
- Hermsdorfer Institut für Technische Keramik e,V,, Hermsdorf/Thür, DE

- Micromeritics GmbH, Möchengladbach, 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/48/93 (1994)	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 (Determination of the pore volume distribution and the specific surface area of solids by mercury intrusion)

Note:
BAM-project "Porous Reference Materials: P. Klobes
The overall co-ordination leading to this certificate and the issuance of the material was performed by B. Röhl-Kuhn.
Statistics: Jörg Polzehl, Weierstraß-Institut Berlin

LEGAL NOTICE

Date of certification: 2005-02-11
Date of shipment: ,,,,,,,,,,,,,,,,,,,,,,,,

BAM
for certified true copy

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Annex
Discrete values of the reference curve with simultaneous prediction bands

Data point No,	Pressure MPa	Low 0,95	Low 0,9	Low 0,8	Reference Curve V_p (mm^3g^{-1})	Up 0,8	Up 0,9	Up 0,95
1	0,24	0	0	0	4,3334	10,791	10,878	10,982
2	0,25	0	0	0	4,6364	10,791	10,878	10,982
3	0,26	0	0	0	4,6364	10,791	10,878	10,982
4	0,27	0	0	0	4,6364	10,791	10,878	10,982
5	0,28	0	0	0	4,6364	10,791	10,878	10,982
6	0,29	0	0	0	4,6771	10,791	10,878	10,982
7	0,3	0	0	0	4,6771	10,791	10,878	10,982
8	0,31	0	0	0	4,6771	10,811	10,898	11,004
9	0,32	0	0	0	4,6771	10,914	11,002	11,107
10	0,33	0	0	0	4,6771	10,914	11,002	11,107
11	0,34	0	0	0	4,6771	10,914	11,002	11,107
12	0,35	0	0	0	4,6771	11,069	11,195	11,336
13	0,36	0	0	0	4,6827	11,179	11,312	11,535
14	0,37	0	0	0	5,0699	12,291	12,546	12,924
15	0,38	0	0	0	5,5452	14,477	15,314	16,519
16	0,39	0	0	0	6,5764	19,451	20,781	22,105
17	0,4	0	0	0	9,0147	24,673	27,524	31,699
18	0,41	0	0	0	11,146	37,777	40,336	42,449
19	0,42	0	0,1827	0,7899	17,697	44,535	48,387	55,188
20	0,43	1,4679	1,5206	1,5337	22,277	61,488	67,448	73,652
21	0,44	1,5211	1,8262	2,656	29,853	75,627	80,238	85,863
22	0,45	5,1553	7,8987	10,524	49,238	86,098	88,095	90,58
23	0,46	13,708	17,33	21,16	61,706	89,829	92,472	94,61
24	0,47	25,406	30,759	35,371	71,594	93,119	95,059	99,3
25	0,48	38,744	43,924	48,323	79,179	97,94	102,83	109,5
26	0,49	49,326	53,295	58,241	84,881	107,46	110,45	113,32
27	0,5	59,438	64,819	68,485	90,224	111,06	113,63	116,93
28	0,51	68,346	72,007	75,787	95,211	114,39	117,19	120,44
29	0,52	75,312	79,991	83,541	99,631	117,7	120,69	124,25
30	0,53	82,346	85,217	86,932	103,02	121,15	124,56	127,28
31	0,54	85,336	86,433	87,777	106,29	124,71	126,49	129,7
32	0,55	86,042	89,068	92,019	109,77	126,36	129,15	132,1
33	0,56	90,002	93,147	95,838	112,64	129,05	131,09	133,66
34	0,57	93,621	96,422	99,032	115,48	130,39	132,83	135,92
35	0,58	96,681	99,494	102,31	117,95	132,43	134,96	138,24
36	0,59	99,732	102,49	104,89	120,32	134,5	134,17	140,39
37	0,6	102,22	105,01	106,7	122,49	122,49	136,55	142,41
38	0,61	104,28	107,02	110,03	124,26	138,54	141,16	143,65
39	0,62	107,01	110,01	112,22	126,37	140,33	142,3	144,57
40	0,63	109,43	111,89	114,16	128,11	141,35	143,18	145,64
41	0,64	111,17	113,82	116,57	129,86	142,13	144,17	146,98
42	0,65	113,18	116,29	118,38	131,19	143,14	145,47	148,58
43	0,66	115,41	117,89	120,19	132,88	144,41	147,01	149,92
44	0,67	117,01	119,59	121,67	133,59	145,9	148,37	151,3
45	0,68	118,55	121,03	123,16	135,43	147,21	149,65	152,03

Data point No,	Pressure MPa	Low 0,95	Low 0,9	Low 0,8	Reference Curve V_p (mm^3g^{-1})	Up 0,8	Up 0,9	Up 0,95
46	0,69	119,97	122,41	124,36	136,54	148,43	150,3	152,87
47	0,7	121,26	123,54	125,37	137,78	149,07	151,22	153,92
48	0,71	122,34	124,41	126,44	138,73	149,97	152,1	155
49	0,72	123,15	125,59	128,06	139,79	150,82	153,47	155,05
50	0,73	124,34	127,29	129,95	140,7	152,19	153,53	155,84
51	0,74	125,97	129,07	131,31	141,61	152,3	154,03	156,88
52	0,75	127,68	130,3	132,39	142,5	152,64	154,96	157,71
53	0,76	128,92	131,33	133,15	143,2	153,54	155,78	158,34
54	0,77	129,92	132,06	133,95	144,28	154,45	156,53	159,02
55	0,78	130,64	132,82	134,49	144,71	155,21	157,38	159,17
56	0,79	131,38	133,37	135,31	145,38	155,87	157,55	160,05
57	0,8	131,86	134,16	135,91	146,07	156,25	157,98	160,95
58	0,81	132,68	134,75	136,8	146,62	156,44	158,79	161,05
59	0,82	133,17	135,64	137,65	147,35	157,31	159,52	161,07
60	0,83	134,2	136,36	138,2	147,8	158,16	159,52	161,49
61	0,84	134,7	137	138,23	148,47	158,25	159,62	161,68
62	0,85	135,5	137,05	138,87	148,69	158,25	160,05	162,09
63	0,86	135,63	137,51	140,13	149,52	158,61	160,18	162,37
64	0,87	135,77	138,78	140,82	149,79	158,86	160,58	162,72
65	0,88	137,06	139,53	141,24	150,27	159,16	160,9	163,11
66	0,89	137,89	139,95	141,59	150,62	159,38	161,21	163,24
67	0,9	138,35	140,36	141,85	150,93	159,76	161,59	163,76
68	0,91	138,77	140,55	142,04	151,39	160,07	161,71	163,8
69	0,92	138,96	140,72	142,58	151,76	160,37	162,21	164,05
70	0,93	139,24	141,33	143	152,16	160,65	162,25	164,13
71	0,94	139,56	141,55	143,66	152,67	160,92	162,49	164,33
72	0,95	139,93	142,27	143,98	152,67	161,08	162,55	164,48
73	0,96	140,47	142,72	143,98	153,04	161,19	162,77	164,64
74	0,97	141,03	142,76	143,99	153,31	161,34	162,88	164,8
75	0,98	141,28	142,76	144,67	153,5	161,47	163,01	165,06
76	0,99	141,28	142,98	145,28	153,78	161,62	163,23	165,29
77	1	141,28	143,84	145,92	153,96	161,77	163,39	165,29
76	1,01	141,95	144,45	146,24	154,26	161,93	163,71	165,44
77	1,02	142,49	144,86	146,49	154,32	162,14	163,71	165,48
78	1,03	143,14	145,14	146,89	154,72	162,41	163,77	165,51
79	1,04	143,44	145,45	147,02	154,78	162,41	163,9	165,52
80	1,05	143,69	145,7	147,33	155,08	162,48	163,94	165,8
81	1,06	144,05	145,97	147,37	155,25	162,59	163,94	165,8
82	1,07	144,21	146,07	147,58	155,25	162,63	164,14	165,82
83	1,08	144,52	146,18	147,71	155,48	162,63	164,22	165,83
84	1,09	144,56	146,41	147,87	155,7	162,78	164,23	165,96
85	1,1	144,69	146,51	147,9	155,7	162,91	164,25	166,03
86	1,11	144,9	146,64	148,14	156	162,91	164,25	166,13
87	1,12	145,01	146,64	148,53	156,12	162,94	164,45	166,13
88	1,13	145,12	147,16	148,58	156,12	162,94	164,48	166,26
89	1,14	145,13	147,26	149	156,22	163,12	164,55	166,3
90	1,15	145,67	147,4	149,04	156,38	163,14	164,55	166,4
91	1,16	145,74	147,77	149,08	156,38	163,23	164,71	166,4
92	1,17	145,88	147,77	149,33	156,44	163,23	164,72	166,4

Data point No,	Pressure MPa	Low 0,95	Low 0,9	Low 0,8	Reference Curve V_p (mm^3g^{-1})	Up 0,8	Up 0,9	Up 0,95
93	1,18	146,24	147,85	149,56	156,65	163,37	164,82	166,4
94	1,19	146,24	148,09	149,62	156,7	163,4	164,82	164,4
95	1,2	146,31	148,32	149,64	156,87	163,49	164,82	166,4
96	1,21	146,54	148,34	149,64	156,96	163,5	164,82	166,4
97	1,22	146,76	148,36	149,64	156,96	163,5	164,82	166,4
98	1,23	146,8	148,36	149,84	157,04	163,5	164,82	166,4
99	1,24	146,82	148,36	150,21	157,15	163,5	164,82	166,4
100	1,25	146,82	148,58	150,21	157,15	163,5	164,82	166,4
101	1,26	146,82	148,93	150,28	157,32	163,5	164,82	166,4
102	1,27	146,97	148,93	150,51	157,35	163,5	164,82	166,4
103	1,28	147,36	149	150,59	157,4	163,5	164,82	166,4
104	1,29	147,39	149,22	150,79	157,49	163,5	164,82	166,45
105	1,3	147,4	149,29	150,79	157,49	163,5	164,82	166,76
106	1,31	147,66	149,5	150,83	157,65	163,5	164,82	166,76
107	1,32	147,7	149,51	151,05	157,65	163,5	164,82	166,76
108	1,33	147,91	149,53	151,09	157,77	163,5	165,16	166,76
109	1,34	147,96	149,74	151,14	157,77	163,5	165,17	166,76
110	1,35	147,96	149,8	151,3	157,81	163,52	165,17	166,76
111	1,36	148,11	149,83	151,31	157,82	163,86	165,17	166,76
112	1,37	148,25	150,01	151,34	157,82	163,86	165,17	166,76
113	1,38	148,25	150,02	151,52	157,86	163,86	165,17	166,76
114	1,39	148,37	150,03	151,53	157,88	163,86	165,17	166,76
115	1,4	148,47	150,23	151,7	157,92	163,86	165,17	166,76
116	1,41	148,47	150,23	151,71	157,92	163,86	165,17	166,76
117	1,42	148,56	150,37	151,72	158,06	163,86	165,17	166,76
118	1,43	148,67	150,42	151,87	158,06	163,86	165,17	166,76
119	1,44	148,69	150,42	151,87	158,06	163,86	165,17	166,76
120	1,45	148,86	150,55	152,05	158,06	163,86	165,17	166,76
121	1,46	148,86	150,57	152,13	158,06	163,86	165,17	166,76
122	1,47	148,87	150,62	152,13	158,06	163,86	165,17	166,76
123	1,48	149,01	150,83	152,13	158,06	163,86	165,17	166,76
124	1,49	149,01	150,83	152,13	158,06	163,86	165,17	166,76
125	1,5	149,14	150,83	152,13	158,06	163,86	165,17	166,76
126	1,51	149,27	150,83	152,13	158,07	163,86	165,17	166,76
127	1,52	149,27	150,83	152,13	158,08	163,86	165,17	166,76
128	1,53	149,27	150,83	152,13	158,11	163,86	165,17	166,76
129	1,54	149,27	150,83	152,13	158,11	163,86	165,17	166,76
130	1,55	149,27	150,83	152,13	158,11	163,86	165,17	166,76

Reference curve : certified pressure-volume curve
Low 0,8; 0,9; 0,95: lower bounding curve of prediction band with significance level 0,8; 0,9; 0,95
Up 0,8; 0,9; 0,95 : upper bounding curve of prediction band with significance level 0,8; 0,9; 0,95