

Federal Institute for Materials Research and Testing

**CERTIFIED REFERENCE MATERIAL
FOR MERCURY INTRUSION**

BAM-P126
Material: Flat Membrane

Certified properties:

Pressure-volume curve between 0,55 MPa and 2,1 MPa of mercury intrusion

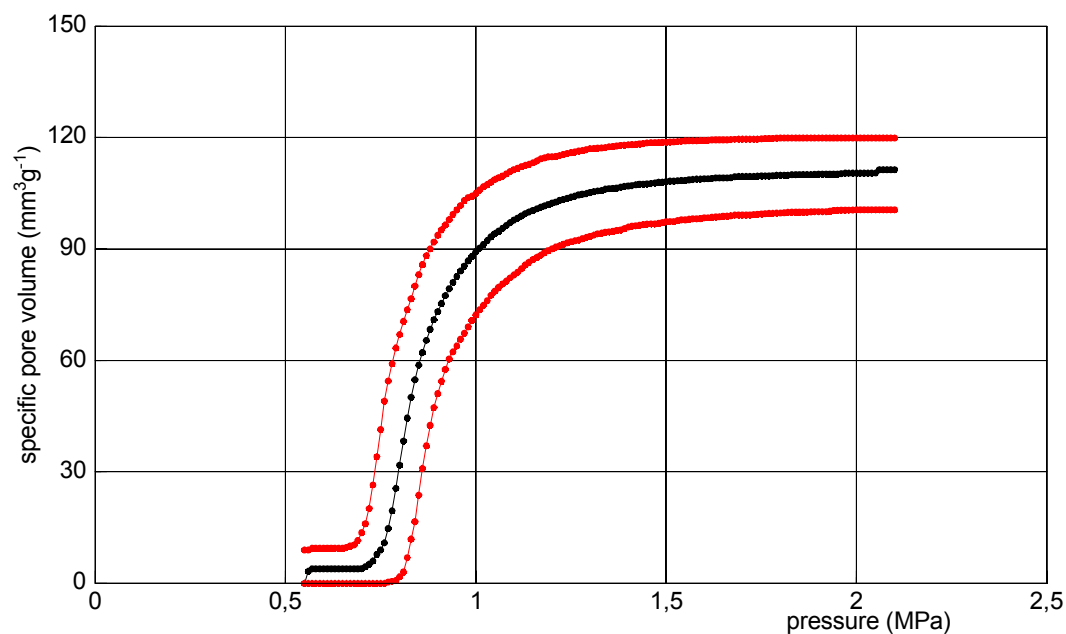


Figure 1 Reference curve (black) with simultaneous prediction band at the significance level 0,95 (red) for the material BAM-P126 (for discrete values see annex)

Certificate

Certified discrete values of BAM-P126 (curve characteristics)

Quantity	Certified values	0,95-Confidence Interval	Unit
y_1 $V_{p, 2,1 \text{ MPa}}$ (specific pore volume at 2,1 MPa)	110,9	110,2 - 111,6	mm^3g^{-1}
y_2	0,8682	0,8648 - 0,8717	MPa
y_3	0,2965	0,2939 - 0,2991	MPa
p_{50}	0,8441	0,8406 - 0,8475	MPa
d_{50}	1,746	1,739 - 1,753	μm

Quantity	Certified values	0,95-Prediction Interval	Unit
y_1 $V_{p, 2,1 \text{ MPa}}$ (specific pore volume at 2,1 MPa)	110,9	102,4 - 119,4	mm^3g^{-1}
y_2	0,8682	0,8274 - 0,9091	MPa
y_3	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

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 2,1 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 $h = 0,05 \text{ MPa}$)

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 $d_p = -4 \gamma \cos\theta / p_{\text{Hg}}$ (assuming a cylindric pore model) has to be carried out using the following values of the parameters: $\gamma = 0.48 \text{ N m}^{-1}$ (surface tension of mercury) and $\theta = 140^\circ$ (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} .

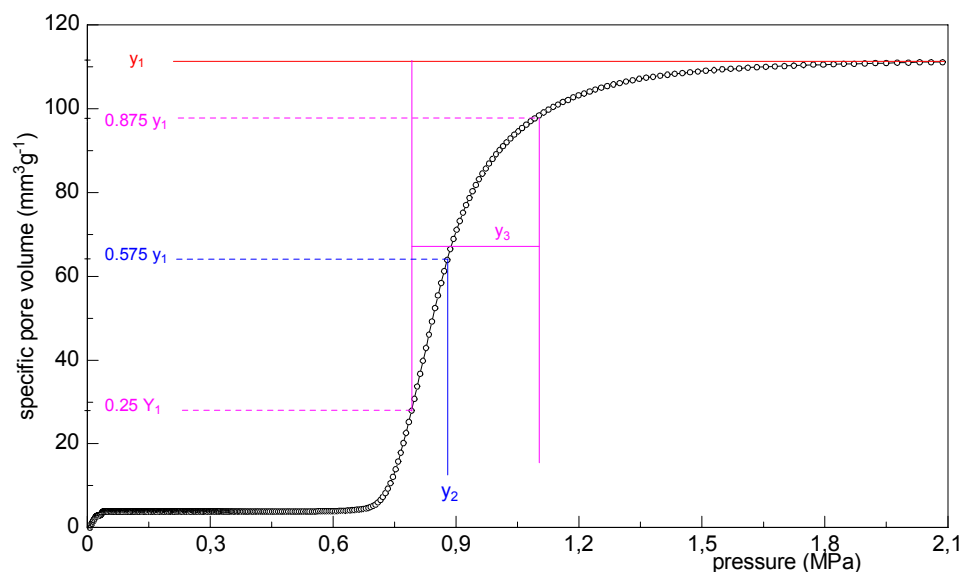


Figure 2 Standardization of the pressure-volume curves (see certification report)

Non-certified properties*:

Quantity	Unit	Value
Specific surface area	$\text{m}^2 \text{g}^{-1}$	0,3
Bulk density	g cm^{-3}	2,6
Apparent density	g cm^{-3}	3,8
Porosity	%	30

* 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 as 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 0,55 and 2,1 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 % (out gassed) 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 diagram with the reference curve and the prediction band, 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 $(1-\alpha)$.
- Definition of the prediction band:
A prediction band of level $(1-\alpha)$ covers the measured curve over the given pressure interval (0,55–2,1 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.

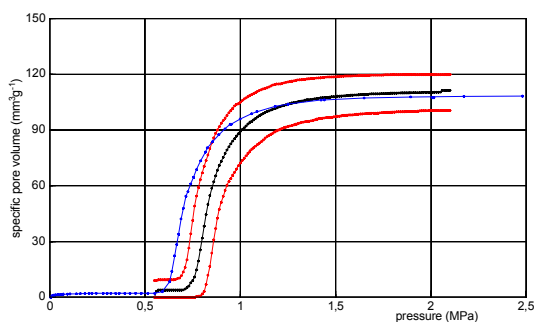


Figure 3 Demonstration of the calibration error in the pressure sensor of the device
reference curve - black
prediction band with 0,95 significance level - red
test curve - blue

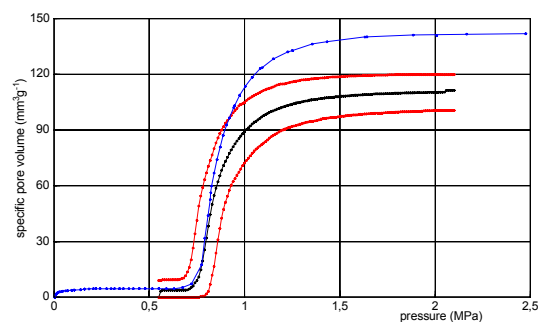


Figure 4 Demonstration of the calibration error of the volume of the device
reference curve - black
prediction band with 0,95 significance level - red
test curve - blue

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
- 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

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This certificate is valid 10 years after seal affixation; this validity may be extended as further evidence of stability becomes available.

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

BAM
for certified true copy

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Analytical Chemistry;
Reference Materials

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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 ³ g ⁻¹)	Up 0,8	Up 0,9	Up 0,95
1	0,55	0	0	0	0	8,6825	9,1201	9,0209
2	0,56	0	0	0	3,2587	8,6825	9,1201	9,0271
3	0,57	0	0	0	3,909	9,1171	9,2705	9,4403
4	0,58	0	0	0	3,909	9,1783	9,3058	9,4564
5	0,59	0	0	0	3,909	9,1783	9,3058	9,4564
6	0,6	0	0	0	3,909	9,1783	9,3058	9,4564
7	0,61	0	0	0	3,909	9,1783	9,3058	9,4564
8	0,62	0	0	0	3,909	9,1783	9,3058	9,4564
9	0,63	0	0	0	3,909	9,1783	9,3058	9,4564
10	0,64	0	0	0	3,909	9,1783	9,3058	9,4564
11	0,65	0	0	0	3,909	9,1783	9,3058	9,4564
12	0,66	0	0	0	3,909	9,1811	9,3854	9,6959
13	0,67	0	0	0	3,909	9,5278	9,7841	10,069
14	0,68	0	0	0	3,909	9,818	10,004	10,355
15	0,69	0	0	0	3,909	10,143	10,635	11,547
16	0,7	0	0	0	3,9653	11,448	12,446	13,655
17	0,71	0	0	0	4,4867	13,471	14,477	16,078
18	0,72	0	0	0	5,2276	15,71	17,479	20,196
19	0,73	0	0	0	6,0463	19,673	22,414	26,504
20	0,74	0	0	0	7,8077	25,394	29,31	34,133
21	0,75	0	0	0	9,0395	32,678	36,469	41,413
22	0,76	0	0,1427	0,3687	10,938	39,302	43,661	49,083
23	0,77	0,3921	0,5696	0,5852	14,788	46,655	50,669	54,483
24	0,78	0,5644	0,5696	0,812	19,541	52,148	55,153	59,119
25	0,79	0,7685	1,2738	1,8964	25,589	56,345	59,629	63,363
26	0,8	1,7943	2,5233	3,4496	31,847	60,525	63,582	66,997
27	0,81	3,1293	5,0947	7,7409	38,338	64,069	66,931	70,54
28	0,82	6,9494	10,272	12,876	44,502	67,376	70,291	73,668
29	0,83	11,92	14,904	18,283	50,1	70,44	73,271	76,659
30	0,84	16,654	21,371	26,147	54,807	73,272	76,165	80,05
31	0,85	23,793	29,169	33,527	58,788	76,24	79,492	83,123
32	0,86	30,948	35,741	39,836	62,173	79,334	82,312	85,819
33	0,87	37,058	41,66	45,575	65,415	81,994	84,901	88,248
34	0,88	42,58	46,908	50,297	68,405	84,492	87,153	90,055
35	0,89	47,333	51,018	54,058	70,99	86,46	88,873	91,914
36	0,9	51,092	54,514	57,517	73,189	88,147	90,679	93,724
37	0,91	54,403	57,873	60,846	75,357	89,873	92,415	95,203
38	0,92	57,611	60,952	63,428	77,465	91,493	93,808	96,475
39	0,93	60,416	63,114	65,196	79,342	92,808	95,013	97,941
40	0,94	62,346	64,752	67,003	81,044	94,004	96,456	99,342
41	0,95	63,927	66,616	68,871	82,624	95,401	97,796	100,56
42	0,96	65,766	68,315	70,595	84,101	96,699	98,976	101,72
43	0,97	67,352	70,053	72,505	85,449	97,82	100,09	103,07
44	0,98	69,057	71,896	74,209	86,821	98,905	101,38	103,88
45	0,99	70,841	73,461	75,616	88,046	100,17	102,24	104,24

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 ³ g ⁻¹)	Up 0,8	Up 0,9	Up 0,95
46	1	72,309	74,844	77,005	89,316	100,95	102,58	104,98
47	1,01	73,622	76,144	78,29	90,31	101,26	103,24	105,93
48	1,02	74,872	77,391	79,802	91,24	101,93	104,15	106,8
49	1,03	76,114	78,88	81,059	92,265	102,8	105,03	107,39
50	1,04	77,55	80,062	82,193	93,304	103,67	105,63	108,02
51	1,05	78,658	81,171	83,153	94,172	104,24	106,21	108,73
52	1,06	79,74	82,041	83,992	94,824	104,81	106,91	109,26
53	1,07	80,585	82,872	84,91	95,576	105,47	107,47	109,67
54	1,08	81,378	83,775	85,755	96,365	106,04	107,86	110,26
55	1,09	82,254	84,578	86,559	97,124	106,43	108,38	110,9
56	1,1	83,04	85,361	87,549	97,816	106,9	108,99	111,42
57	1,11	83,781	86,346	88,614	98,35	107,49	109,55	111,76
58	1,12	84,704	87,375	89,409	98,964	108,07	109,92	112,2
59	1,13	85,731	88,137	90,113	99,563	108,45	110,3	112,54
60	1,14	86,484	88,813	90,753	99,997	108,78	110,7	112,83
61	1,15	87,15	89,442	91,375	100,37	109,2	110,95	113,23
62	1,16	87,77	90,047	92,092	100,83	109,46	111,32	113,72
63	1,17	88,336	90,748	92,646	101,28	109,79	111,72	114,27
64	1,18	89,009	91,281	93,189	101,65	110,15	112,29	114,58
65	1,19	89,569	91,805	93,648	101,97	110,66	112,69	114,82
66	1,2	90,056	92,271	94,052	102,35	111,13	112,94	114,86
67	1,21	90,546	92,648	94,492	102,67	111,39	113,11	114,92
68	1,22	90,896	93,087	94,837	103,11	111,62	113,11	115,17
69	1,23	91,335	93,427	95,165	103,34	111,65	113,24	115,46
70	1,24	91,679	93,755	95,363	103,67	111,7	113,52	115,75
71	1,25	92,007	93,953	95,652	103,97	111,95	113,79	115,95
72	1,26	92,24	94,213	96,039	104,27	112,21	114,05	116,17
73	1,27	92,443	94,586	96,358	104,55	112,48	114,24	116,36
74	1,28	92,778	94,911	96,713	104,78	112,67	114,45	116,53
75	1,29	93,12	95,228	97,119	104,94	112,88	114,61	116,77
76	1,3	93,408	95,638	97,338	105,19	113,06	114,79	117,02
77	1,31	93,782	95,89	97,58	105,49	113,22	115,05	117,09
78	1,32	94,117	96,102	97,749	105,64	113,43	115,25	117,18
79	1,33	94,291	96,318	97,82	105,76	113,68	115,31	117,24
80	1,34	94,536	96,369	98,097	106,1	113,8	115,39	117,34
81	1,35	94,652	96,591	98,264	106,24	113,85	115,44	117,53
82	1,36	94,747	96,79	98,601	106,35	113,93	115,56	117,63
83	1,37	95,015	97,013	99,043	106,41	113,98	115,74	117,77
84	1,38	95,155	97,469	99,33	106,62	114,14	115,83	117,92
85	1,39	95,503	97,804	99,46	106,89	114,29	115,97	117,98
86	1,4	95,912	97,996	99,552	107,02	114,36	116,11	118,04
87	1,41	96,174	98,064	99,761	107,13	114,52	116,18	118,11
88	1,42	96,293	98,233	99,942	107,29	114,64	116,23	118,15
89	1,43	96,38	98,435	99,995	107,48	114,69	116,3	118,31
90	1,44	96,573	98,552	99,995	107,48	114,74	116,33	118,44
91	1,45	96,752	98,553	100,1	107,58	114,81	116,48	118,54

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 ³ g ⁻¹)	Up 0,8	Up 0,9	Up 0,95
92	1,46	96,819	98,577	100,39	107,66	114,85	116,62	118,64
93	1,47	96,819	98,792	100,61	107,77	115,02	116,71	118,64
94	1,48	96,872	99,081	100,64	107,99	115,13	116,81	118,68
95	1,49	97,13	99,081	100,7	108,01	115,23	116,82	118,71
96	1,5	97,383	99,187	100,98	108,17	115,31	116,86	118,76
97	1,51	97,442	99,367	101,13	108,26	115,31	116,88	118,82
98	1,52	97,443	99,639	101,15	108,26	115,35	116,92	118,89
99	1,53	97,666	99,674	101,29	108,39	115,38	116,98	118,97
100	1,54	97,907	99,741	101,36	108,45	115,42	117,04	119,02
101	1,55	97,914	99,883	101,38	108,48	115,47	117,13	119,12
102	1,56	97,999	99,905	101,57	108,66	115,53	117,17	119,15
103	1,57	98,134	99,965	101,7	108,75	115,62	117,26	119,15
104	1,58	98,135	100,16	101,72	108,81	115,66	117,32	119,16
105	1,59	98,217	100,23	101,85	108,81	115,75	117,32	119,23
106	1,6	98,413	100,29	101,92	108,9	115,81	117,32	119,25
107	1,61	98,461	100,41	102,04	108,92	115,81	117,37	119,27
108	1,62	98,525	100,48	102,13	109,03	115,81	117,42	119,39
109	1,63	98,641	100,61	102,21	109,17	115,85	117,42	119,42
110	1,64	98,701	100,66	102,36	109,17	115,9	117,5	119,42
111	1,65	98,837	100,79	102,39	109,17	115,9	117,59	119,42
112	1,66	98,89	100,9	102,39	109,18	115,96	117,59	119,43
113	1,67	99,003	100,91	102,39	109,27	116,06	117,59	119,48
114	1,68	99,122	100,91	102,41	109,48	116,07	117,59	119,54
115	1,69	99,13	100,91	102,52	109,55	116,07	117,62	119,58
116	1,7	99,13	100,94	102,63	109,55	116,07	117,67	119,58
117	1,71	99,13	101,06	102,72	109,55	116,09	117,72	119,58
118	1,72	99,155	101,17	102,83	109,55	116,14	117,75	119,58
119	1,73	99,271	101,25	102,89	109,55	116,19	117,75	119,58
120	1,74	99,369	101,36	102,89	109,64	116,23	117,75	119,58
121	1,75	99,443	101,4	102,94	109,69	116,23	117,75	119,66
122	1,76	99,55	101,41	102,97	109,69	116,23	117,75	119,78
123	1,77	99,616	101,46	103,06	109,69	116,23	117,75	119,79
124	1,78	99,616	101,49	103,2	109,78	116,23	117,86	119,79
125	1,79	99,657	101,58	103,23	109,89	116,23	117,95	119,84
126	1,8	99,691	101,71	103,23	109,89	116,28	117,95	119,91
127	1,81	99,751	101,74	103,23	109,89	116,4	117,95	119,91
128	1,82	99,893	101,74	103,23	109,89	116,43	118,02	119,91
129	1,83	99,949	101,74	103,28	109,99	116,43	118,08	119,91
130	1,84	99,949	101,74	103,37	110,01	116,46	118,08	119,91
131	1,85	99,949	101,78	103,39	110,04	116,54	118,08	119,91
132	1,86	99,949	101,87	103,39	110,06	116,55	118,08	119,91
133	1,87	99,961	101,9	103,39	110,06	116,55	118,08	119,91
134	1,88	100,04	101,9	103,4	110,06	116,55	118,08	119,91
135	1,89	100,1	101,9	103,56	110,09	116,55	118,08	119,91
136	1,9	100,1	101,91	103,75	110,19	116,55	118,08	119,91
137	1,91	100,1	102,07	103,77	110,19	116,55	118,08	119,91

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 ³ g ⁻¹)	Up 0,8	Up 0,9	Up 0,95
138	1,92	100,1	102,24	103,78	110,19	116,55	118,08	119,91
139	1,93	100,16	102,27	103,79	110,19	116,55	118,08	119,91
140	1,94	100,38	102,28	103,83	110,19	116,55	118,08	119,91
141	1,95	100,46	102,29	103,89	110,19	116,55	118,08	119,91
142	1,96	100,47	102,32	103,91	110,32	116,55	118,08	119,91
143	1,97	100,47	102,38	103,91	110,39	116,55	118,08	119,91
144	1,98	100,49	102,41	103,91	110,39	116,55	118,08	119,91
145	1,99	100,54	102,41	103,91	110,39	116,55	118,08	119,91
146	2,0	100,6	102,41	103,91	110,39	116,55	118,08	119,91
147	2,01	100,61	102,41	103,91	110,39	116,55	118,08	119,91
148	2,02	100,61	102,41	103,91	110,42	116,55	118,08	119,91
149	2,03	100,61	102,41	103,91	110,43	116,55	118,08	119,91
150	2,04	100,61	102,41	103,91	110,43	116,55	118,08	119,91
151	2,05	100,61	102,41	103,93	110,47	116,55	118,08	119,91
152	2,06	100,61	102,41	104,09	111,41	116,55	118,08	119,91
153	2,07	100,61	102,41	104,17	111,41	116,55	118,08	119,91
154	2,08	100,61	102,52	104,17	111,41	116,55	118,08	119,91
155	2,09	100,61	102,67	104,17	111,41	116,55	118,08	119,91
156	2,1	100,61	102,67	104,21	111,41	116,55	118,08	119,91

Reference curve
 Low 0,8; 0,9; 0,95
 Up 0,8; 0,9; 0,95

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