CERTIFICATE OF ANALYSIS

ERM®-FD123

Ceramic filter tubes

Certified Values
Mercury intrusion curve between 0.28 MPa and 1.41 MPa

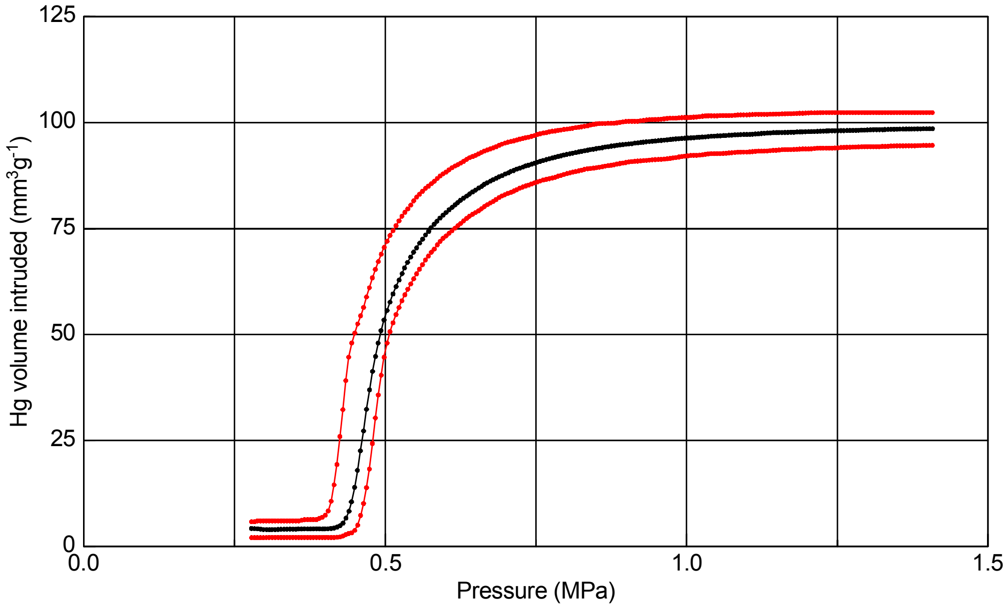

Figure 1: Reference curve (black) with simultaneous prediction bands at the significance level 0.95 (for discrete values see annex)

Pressure-volume curve characteristics			
Quantity	Certified value [1)]	Uncertainty U [2)]	Unit
y_1 [3)]	99.52	± 3.44	mm^3g^{-1}
y_2 [4)]	0.4966	± 0.0180	MPa
y_3 [5)]	0.2151	± 0.0156	MPa
p_{50}	0.4829	± 0.0239	MPa
d_{50}	3.0520	± 0.1533	µm

1) Pressure volume curves from designed round robins are analysed by means of a multivariate variance components model for the curves characteristics y_1, y_2 and y_3. The results are mean curve characteristics (certified values) and confidence intervals for the curve characteristics. Adjusted curves and statistics from the variance components model are used to create a certified pressure volume curve with confidence bands and prediction bands.

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.

3) y_1: Intruded volume at the saturation point 1.41 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 = 0.035 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).

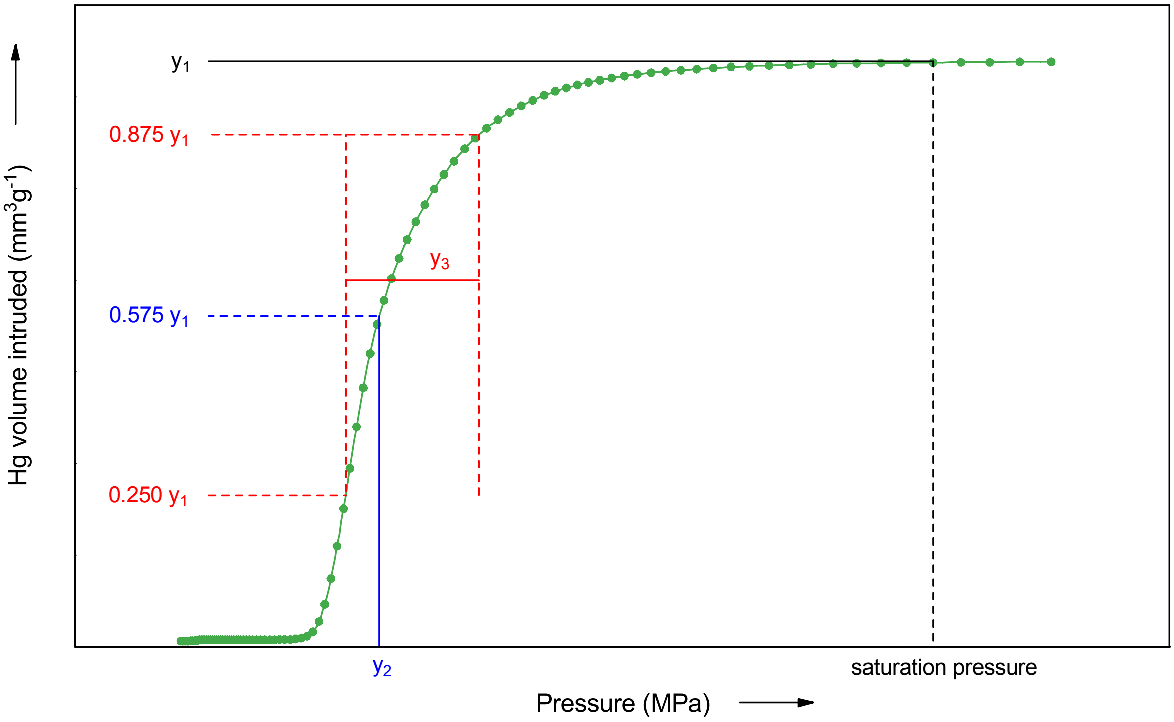

Figure 2: Definitions of the pressure-volume curve characteristics y_1, y_2, and y_3 (for further information please see the certification report).

NOTE

European Reference Material ERM®-FD123 was originally certified as BAM-P123. It was produced and certified under the responsibility of BAM Bundesanstalt für Materialforschung und -prüfung according to the principles laid down in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-LGC-IRMM. Information on these guidelines is available on the Internet (http://www.erm-crm.org).

Accepted as an ERM®, Berlin, 2004-04-14.

This certificate is valid for three years after purchase.

Sales date:

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Additional Material Information		
Quantity	Informative value	Unit
Specific surface area	0.2	$m^2\ g^{-1}$
Bulk density	2.8	$g\ cm^{-3}$
Apparent density	3.98	$g\ cm^{-3}$
Porosity	29	%

DESCRIPTION OF THE SAMPLE

The reference material is intended for checking the performance of mercury porosimeters in the low-pressure range between 0.28 and 1.41 MPa.

The reference material consists of filter tube pieces of α-alumina produced by the Institut für Technische Keramik, e.V. (HITK), Hermsdorf, Germany. The whole batch of the material was divided into 75 tubes. Each tube was cut into 20 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.

ANALYTICAL METHOD USED FOR CERTIFICATION

Mercury intrusion according to DIN 66133

PARTICIPANTS

Co-ordination
BAM Bundesanstalt für Materialforschung und -prüfung, DE

Participants:

- Amtliche Materialprüfanstalt für das Bauwesen, TU Braunschweig, DE
- BAM Bundesanstalt für Materialforschung und -prüfung, (5 instruments in 2 laboratories), Berlin, DE
- Degussa AG, Hanau, DE
- Delft University of Technology, Delft, NL
- DMT - Gesellschaft für Lehre und Bildung mbH, Bochum, DE
- Dr. C. Otto Feuerfest GmbH, Bochum, DE
- Eidgenössische Materialprüfungs- und Forschungsanstalt (EMPA), Dübendorf, CH
- Engelhard De Meern B. V., De Meern, NL
- Forschungsinstitut der Zementindustrie Düsseldorf, Düsseldorf, DE
- Forschungsinstitut für anorganische Werkstoffe - Glas/Keramik - GmbH, Höhr-Grenzhausen, DE
- Fraunhofer-Institut für Bauphysik, Valley, DE
- Grace GmbH, Worms, DE
- Hermsdorfer Institut für Technische Keramik e.V., Hermsdorf/Thür., DE
- Hüls Infracor GmbH, Marl, DE
- Micromeritics GmbH, Mönchengladbach, DE
- Quantachrome GmbH, Odelzhausen, DE
- Rheinisch-Westfälische Technische Hochschule, Aachen, (2 laboratories), DE
- Technische Universität Dresden, Dresden, DE
- Technische Universität Hamburg-Harburg, Hamburg, DE
- ThermoQuest Italia S.p.A., CE Instruments, Rodano (Milan), IT
- Universität der Bundeswehr, München, DE
- Universität Gesamthochschule Kassel, DE
- Universität Hannover, Hannover, DE
- Universität Karlsruhe, Karlsruhe, DE

INSTRUCTIONS FOR USE

The recommended sample intake is one piece filter tube per experiment.
Prior to the analysis, a heating procedure for drying the sample is not necessary.
Use mercury with a purity of 99.99 % (outgassed) or better.

INSTRUCTIONS FOR DATA EVALUATION

- Measure one piece of the tubes and plot your measured pressure volume curve in one diagram with the reference curve and the prediction bands (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 of level (1–α) covers the measured curve over the given pressure interval (0.28 to 1.41 MPa) completely with the specified probability. The size of prediction bands depends on the number of measured points per curve. Band given here require about 60 measured points or more 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.

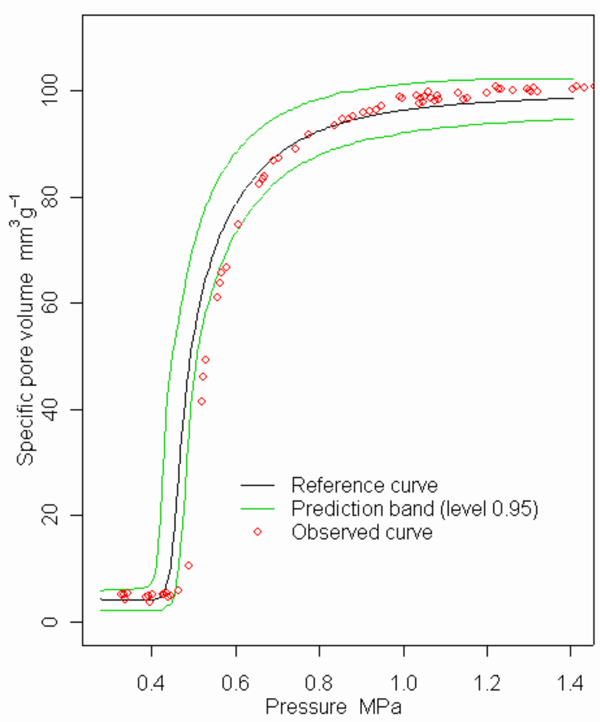

Figure 3: Demonstration of a pressure sensor error of the porosimeter

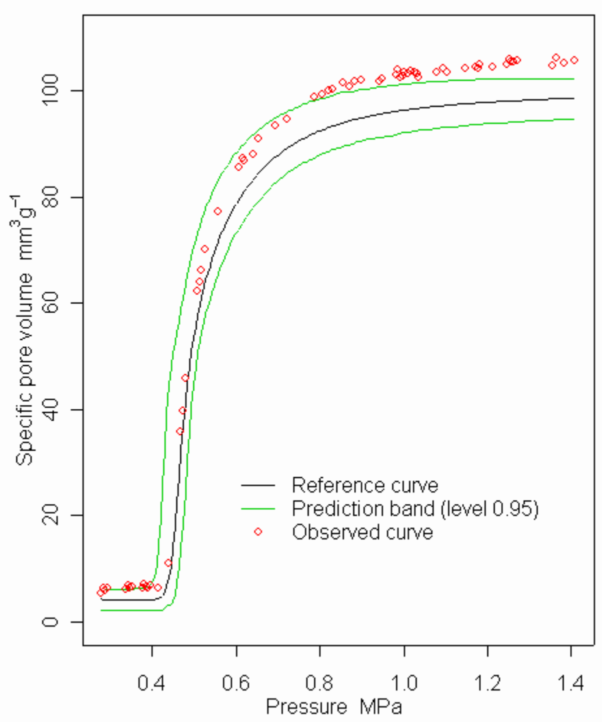

Figure 4: Demonstration of a volume calibration error of the porosimeter

STORAGE

The closed bottle should be stored at ambient temperature in a clean and dry place.

TECHNICAL REPORT

A detailed technical report (in German) describing the analysis procedures and the treatment of the analytical data used to certify ERM®-FD123 is available on request.

REFERENCES

Guidelines for the production and certification of BAM reference materials

BCR/01/97 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)

Supply of Reference Materials by BAM Bundesanstalt für Materialforschung und -prüfung:

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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	0.278	2.062	4.237	5.828
2	0.283	2.062	4.174	5.828
3	0.288	2.062	4.164	5.992
4	0.293	2.062	4.061	6.023
5	0.298	2.062	3.990	6.023
6	0.303	2.076	3.957	6.023
7	0.308	2.086	3.955	6.023
8	0.312	2.086	3.967	6.023
9	0.317	2.086	3.981	6.023
10	0.322	2.086	3.999	6.023
11	0.327	2.086	4.018	6.023
12	0.332	2.086	4.036	6.023
13	0.337	2.086	4.046	6.023
14	0.342	2.086	4.054	6.023
15	0.347	2.086	4.058	6.023
16	0.352	2.086	4.062	6.023
17	0.356	2.086	4.088	6.025
18	0.361	2.086	4.113	6.162
19	0.366	2.086	4.126	6.310
20	0.371	2.086	4.112	6.324
21	0.376	2.086	4.113	6.324
22	0.381	2.086	4.129	6.331
23	0.386	2.086	4.135	6.434
24	0.391	2.086	4.128	6.605
25	0.396	2.086	4.112	6.869
26	0.400	2.086	4.123	7.334
27	0.405	2.086	4.169	8.363
28	0.410	2.087	4.236	10.666
29	0.415	2.098	4.328	14.492
30	0.420	2.123	4.528	19.302
31	0.425	2.216	4.875	25.933
32	0.430	2.447	5.477	32.271
33	0.435	2.793	6.675	39.121
34	0.440	3.030	8.288	44.639
35	0.444	3.193	10.516	47.996
36	0.449	3.745	13.937	50.340
37	0.454	4.974	17.944	52.497
38	0.459	7.306	22.550	54.426
39	0.464	10.109	27.258	56.395
40	0.469	13.861	32.359	58.848

Data Point No.	p_{Hg} (MPa)	$V_{Hg} - U$ (mm^3g^{-1})	V_{Hg} (mm^3g^{-1})	$V_{Hg} + U$ (mm^3g^{-1})
41	0.474	18.233	36.951	61.030
42	0.479	24.294	41.282	63.395
43	0.484	30.283	44.828	65.357
44	0.488	35.727	47.988	67.214
45	0.493	40.395	50.923	68.948
46	0.498	44.609	53.409	70.556
47	0.503	47.978	55.667	72.014
48	0.508	50.674	57.638	73.367
49	0.513	52.742	59.573	74.514
50	0.518	54.689	61.296	75.689
51	0.523	56.362	62.864	76.797
52	0.528	57.955	64.380	77.919
53	0.532	59.383	65.704	78.844
54	0.537	60.696	67.020	79.635
55	0.542	61.925	68.261	80.532
56	0.547	63.143	69.434	81.533
57	0.552	64.315	70.447	82.408
58	0.557	65.407	71.565	83.116
59	0.562	66.485	72.508	83.803
60	0.567	67.553	73.497	84.461
61	0.572	68.496	74.326	84.996
62	0.576	69.403	75.220	85.592
63	0.581	70.174	76.014	86.162
64	0.586	71.137	76.734	86.824
65	0.591	71.980	77.481	87.416
66	0.596	72.741	78.180	87.950
67	0.601	73.265	78.791	88.340
68	0.606	73.870	79.452	88.861
69	0.611	74.474	80.000	89.364
70	0.616	75.078	80.606	89.757
71	0.620	75.676	81.126	90.091
72	0.625	76.223	81.698	90.452
73	0.630	76.833	82.179	90.899
74	0.635	77.411	82.664	91.338
75	0.640	77.947	83.157	91.735
76	0.645	78.358	83.644	91.998
77	0.650	78.799	84.095	92.286
78	0.655	79.178	84.555	92.600
79	0.660	79.652	84.959	92.968
80	0.664	80.176	85.407	93.287

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	0.669	80.651	85.785	93.564
82	0.674	81.027	86.178	93.807
83	0.679	81.413	86.537	94.035
84	0.684	81.850	86.852	94.310
85	0.689	82.318	87.255	94.650
86	0.694	82.709	87.513	94.939
87	0.699	82.999	87.874	95.146
88	0.704	83.318	88.124	95.396
89	0.708	83.531	88.457	95.545
90	0.713	83.864	88.716	95.720
91	0.718	84.186	89.002	95.899
92	0.723	84.543	89.247	96.099
93	0.728	84.803	89.503	96.269
94	0.733	85.049	89.780	96.445
95	0.738	85.253	89.988	96.573
96	0.743	85.518	90.204	96.743
97	0.748	85.761	90.434	96.955
98	0.752	86.003	90.659	97.199
99	0.757	86.236	90.856	97.397
100	0.762	86.466	91.053	97.533
101	0.767	86.627	91.288	97.646
102	0.772	86.763	91.457	97.815
103	0.777	86.935	91.650	97.958
104	0.782	87.105	91.849	98.036
105	0.787	87.354	92.015	98.155
106	0.792	87.528	92.188	98.268
107	0.796	87.771	92.334	98.390
108	0.801	87.987	92.507	98.466
109	0.806	88.151	92.645	98.547
110	0.811	88.316	92.798	98.681
111	0.816	88.458	92.936	98.806
112	0.821	88.625	93.115	98.931
113	0.826	88.772	93.236	99.028
114	0.831	88.961	93.371	99.140
115	0.836	89.102	93.506	99.274
116	0.840	89.202	93.624	99.451
117	0.845	89.276	93.742	99.600
118	0.850	89.363	93.856	99.706
119	0.855	89.488	93.982	99.770
120	0.860	89.614	94.091	99.799
121	0.865	89.740	94.224	99.812
122	0.870	89.832	94.325	99.831
123	0.875	89.933	94.452	99.848
124	0.880	90.068	94.537	99.877
125	0.884	90.233	94.639	99.941
126	0.889	90.359	94.727	100.050
127	0.894	90.461	94.810	100.180
128	0.899	90.581	94.880	100.280
129	0.904	90.709	94.972	100.300
130	0.909	90.821	95.041	100.300
131	0.914	90.887	95.143	100.300
132	0.919	90.925	95.222	100.320
133	0.924	90.969	95.322	100.430
134	0.928	91.032	95.388	100.540
135	0.933	91.100	95.486	100.620
136	0.938	91.186	95.539	100.690
137	0.943	91.251	95.615	100.730
138	0.948	91.293	95.671	100.740
139	0.953	91.309	95.751	100.770
140	0.958	91.344	95.800	100.840
141	0.963	91.392	95.883	100.910
142	0.968	91.477	95.939	101.000
143	0.973	91.560	96.005	101.030
144	0.977	91.669	96.088	101.030
145	0.982	91.792	96.154	101.060
146	0.987	91.900	96.215	101.130
147	0.992	92.001	96.261	101.180
148	0.997	92.109	96.314	101.190
149	1.002	92.181	96.357	101.190
150	1.007	92.256	96.405	101.230
151	1.012	92.270	96.441	101.290
152	1.017	92.274	96.499	101.340
153	1.021	92.314	96.557	101.410
154	1.026	92.411	96.604	101.510
155	1.031	92.542	96.664	101.580
156	1.036	92.616	96.711	101.600
157	1.041	92.664	96.762	101.600
158	1.046	92.684	96.812	101.600
159	1.051	92.688	96.850	101.610
160	1.056	92.702	96.901	101.650

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})
161	1.061	92.741	96.941	101.660
162	1.065	92.783	96.979	101.660
163	1.070	92.832	97.025	101.660
164	1.075	92.902	97.060	101.680
165	1.080	92.971	97.099	101.740
166	1.085	93.028	97.129	101.820
167	1.090	93.054	97.173	101.840
168	1.095	93.068	97.196	101.840
169	1.100	93.079	97.221	101.840
170	1.105	93.112	97.254	101.870
171	1.109	93.169	97.274	101.910
172	1.114	93.240	97.333	101.940
173	1.119	93.271	97.371	101.950
174	1.124	93.274	97.447	101.950
175	1.129	93.280	97.484	101.950
176	1.134	93.330	97.539	101.950
177	1.139	93.405	97.585	101.960
178	1.144	93.460	97.623	101.980
179	1.149	93.485	97.639	102.020
180	1.153	93.523	97.656	102.040
181	1.158	93.601	97.667	102.070
182	1.163	93.647	97.686	102.090
183	1.168	93.665	97.698	102.120
184	1.173	93.669	97.726	102.130
185	1.178	93.684	97.755	102.150
186	1.183	93.718	97.782	102.190
187	1.188	93.763	97.811	102.220
188	1.192	93.791	97.840	102.220
189	1.197	93.796	97.860	102.250
190	1.202	93.797	97.878	102.290
191	1.207	93.819	97.897	102.310
192	1.212	93.886	97.923	102.310
193	1.217	93.956	97.952	102.320
194	1.222	93.980	97.979	102.350
195	1.227	93.981	98.007	102.370
196	1.232	93.981	98.034	102.370

Data Point No.	p_{Hg} (MPa)	$V_{Hg} – U$ (mm^3g^{-1})	V_{Hg} (mm^3g^{-1})	$V_{Hg} + U$ (mm^3g^{-1})
197	1.236	93.981	98.063	102.370
198	1.241	93.984	98.076	102.370
199	1.246	94.010	98.085	102.370
200	1.251	94.073	98.089	102.370
201	1.256	94.128	98.101	102.370
202	1.261	94.158	98.115	102.370
203	1.266	94.171	98.124	102.370
204	1.271	94.192	98.142	102.370
205	1.276	94.219	98.154	102.370
206	1.281	94.230	98.175	102.370
207	1.285	94.267	98.204	102.370
208	1.290	94.295	98.229	102.370
209	1.295	94.320	98.251	102.370
210	1.300	94.323	98.286	102.370
211	1.305	94.323	98.311	102.370
212	1.310	94.323	98.336	102.370
213	1.315	94.324	98.347	102.370
214	1.320	94.337	98.358	102.370
215	1.325	94.370	98.363	102.370
216	1.329	94.398	98.374	102.370
217	1.334	94.415	98.391	102.370
218	1.339	94.458	98.412	102.370
219	1.344	94.499	98.428	102.370
220	1.349	94.516	98.450	102.370
221	1.354	94.516	98.464	102.370
222	1.359	94.519	98.476	102.370
223	1.364	94.531	98.489	102.370
224	1.369	94.540	98.494	102.370
225	1.373	94.540	98.505	102.370
226	1.378	94.542	98.512	102.370
227	1.383	94.553	98.525	102.370
228	1.388	94.609	98.537	102.370
229	1.393	94.626	98.549	102.370
230	1.398	94.627	98.562	102.370
231	1.403	94.627	98.574	102.370
232	1.408	94.644	98.574	102.370

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