CERTIFICATE OF ANALYSIS

ERM®-FD123

Ceramic filter tubes

Certified Values

Mercury intrusion curve between 0.28 MPa and 1.41 MPa

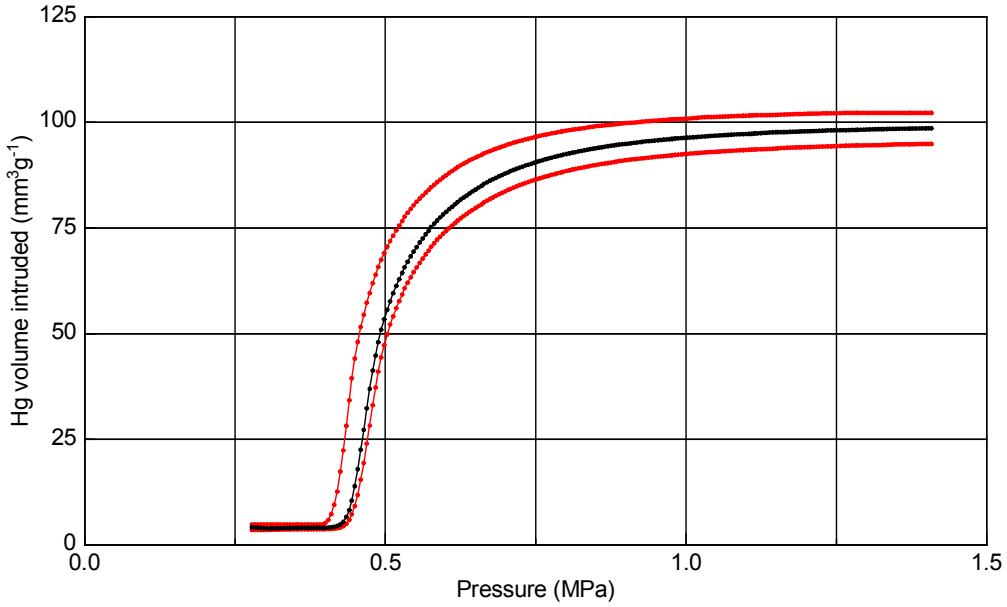

Figure 1: Reference curve (black) with simultaneous confidence bands at the significance level 0.95 (for discrete values see annex)

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.

[2] The confidence 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).

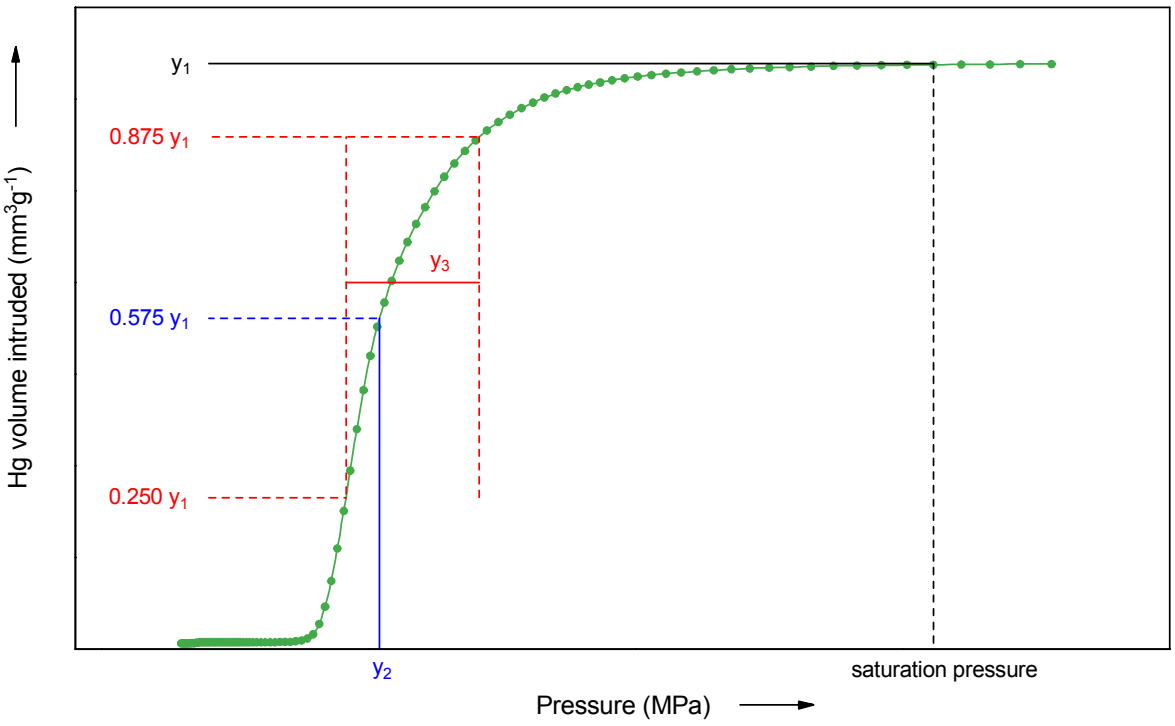

Figure 2: Definitions of the pressure-volume curve characteristics y_1, y_2, and y_3 (for further information please see the certification report).

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
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- 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 confidence 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 confidence band at level (1–α).
- The confidence band is defined as follows:

  A confidence 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 confidence 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.

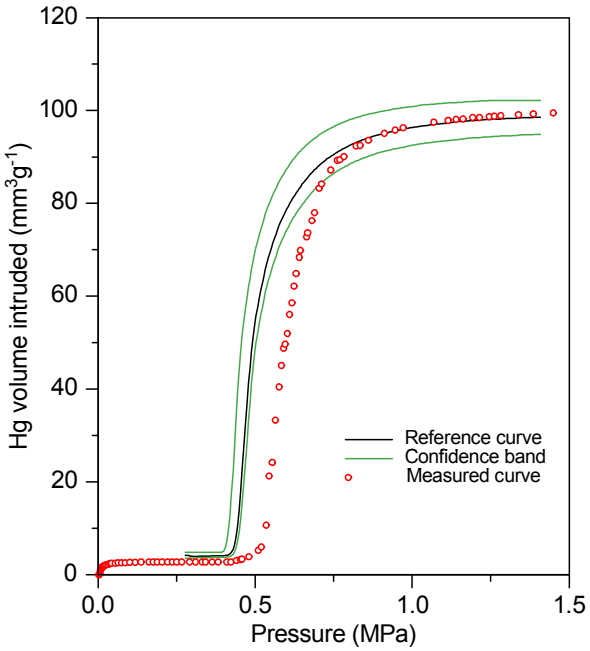

Figure 3: Demonstration of a pressure sensor error of the porosimeter

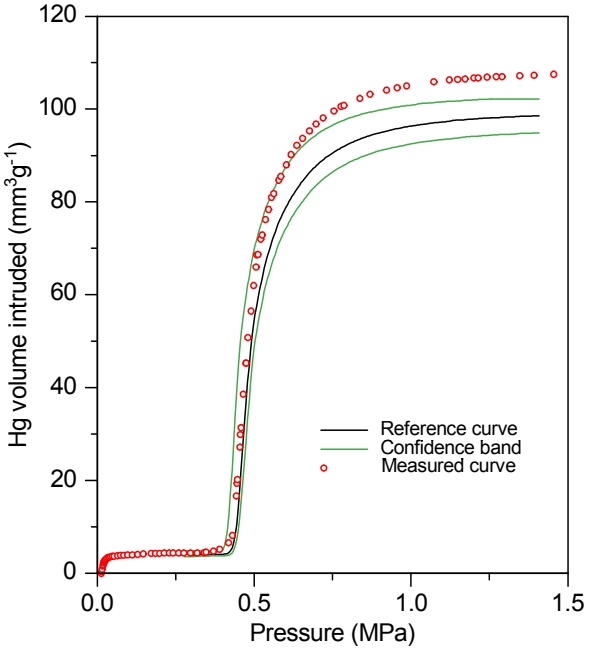

Figure 4: Demonstration of a volume calibration error of the porosimeter

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 confidence 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	3.580	4.237	4.870
2	0.283	3.580	4.174	4.870
3	0.288	3.580	4.164	4.874
4	0.293	3.580	4.061	4.874
5	0.298	3.580	3.990	4.874
6	0.303	3.582	3.957	4.874
7	0.308	3.602	3.955	4.874
8	0.312	3.636	3.967	4.874
9	0.317	3.660	3.981	4.874
10	0.322	3.667	3.999	4.874
11	0.327	3.671	4.018	4.874
12	0.332	3.686	4.036	4.874
13	0.337	3.703	4.046	4.874
14	0.342	3.719	4.054	4.874
15	0.347	3.727	4.058	4.874
16	0.352	3.729	4.062	4.874
17	0.356	3.738	4.088	4.874
18	0.361	3.750	4.113	4.874
19	0.366	3.751	4.126	4.874
20	0.371	3.751	4.112	4.874
21	0.376	3.751	4.113	4.874
22	0.381	3.751	4.129	4.874
23	0.386	3.754	4.135	4.874
24	0.391	3.767	4.128	4.874
25	0.396	3.770	4.112	4.909
26	0.400	3.771	4.123	5.199
27	0.405	3.779	4.169	5.955
28	0.410	3.817	4.236	7.331
29	0.415	3.877	4.328	9.556
30	0.420	3.958	4.528	12.623
31	0.425	4.140	4.875	17.414
32	0.430	4.457	5.477	22.386
33	0.435	5.009	6.675	28.232
34	0.440	5.950	8.288	34.231
35	0.444	7.268	10.516	39.443
36	0.449	9.203	13.937	44.101
37	0.454	11.859	17.944	48.037
38	0.459	15.506	22.550	51.587
39	0.464	19.402	27.258	54.463
40	0.469	24.010	32.359	57.322

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	28.272	36.951	59.590
42	0.479	33.110	41.282	61.967
43	0.484	37.285	44.828	63.938
44	0.488	41.044	47.988	65.762
45	0.493	44.355	50.923	67.455
46	0.498	47.307	53.409	69.051
47	0.503	49.864	55.667	70.532
48	0.508	52.178	57.638	71.937
49	0.513	54.100	59.573	73.154
50	0.518	56.025	61.296	74.423
51	0.523	57.690	62.864	75.556
52	0.528	59.275	64.380	76.649
53	0.532	60.729	65.704	77.667
54	0.537	62.077	67.020	78.613
55	0.542	63.327	68.261	79.504
56	0.547	64.540	69.434	80.380
57	0.552	65.686	70.447	81.212
58	0.557	66.736	71.565	81.948
59	0.562	67.757	72.508	82.655
60	0.567	68.749	73.497	83.353
61	0.572	69.647	74.326	83.984
62	0.576	70.575	75.220	84.646
63	0.581	71.351	76.014	85.207
64	0.586	72.209	76.734	85.824
65	0.591	72.931	77.481	86.379
66	0.596	73.681	78.180	86.959
67	0.601	74.297	78.791	87.445
68	0.606	74.982	79.452	87.993
69	0.611	75.582	80.000	88.479
70	0.616	76.160	80.606	88.940
71	0.620	76.744	81.126	89.389
72	0.625	77.266	81.698	89.791
73	0.630	77.803	82.179	90.208
74	0.635	78.304	82.664	90.597
75	0.640	78.826	83.157	90.992
76	0.645	79.254	83.644	91.320
77	0.650	79.735	84.095	91.677
78	0.655	80.145	84.555	91.988
79	0.660	80.598	84.959	92.322
80	0.664	81.037	85.407	92.638

Annex

Discrete values of the reference curve with simultaneous confidence 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	81.456	85.785	92.935
82	0.674	81.843	86.178	93.205
83	0.679	82.238	86.537	93.470
84	0.684	82.614	86.852	93.733
85	0.689	82.985	87.255	94.002
86	0.694	83.326	87.513	94.255
87	0.699	83.619	87.874	94.471
88	0.704	83.984	88.124	94.754
89	0.708	84.226	88.457	94.938
90	0.713	84.555	88.716	95.172
91	0.718	84.826	89.002	95.368
92	0.723	85.119	89.247	95.571
93	0.728	85.364	89.503	95.743
94	0.733	85.635	89.780	95.938
95	0.738	85.858	89.988	96.099
96	0.743	86.120	90.204	96.289
97	0.748	86.349	90.434	96.464
98	0.752	86.570	90.659	96.647
99	0.757	86.773	90.856	96.809
100	0.762	86.987	91.053	96.962
101	0.767	87.180	91.288	97.097
102	0.772	87.377	91.457	97.249
103	0.777	87.573	91.650	97.391
104	0.782	87.737	91.849	97.502
105	0.787	87.961	92.015	97.662
106	0.792	88.114	92.188	97.778
107	0.796	88.311	92.334	97.920
108	0.801	88.489	92.507	98.037
109	0.806	88.645	92.645	98.137
110	0.811	88.809	92.798	98.247
111	0.816	88.949	92.936	98.337
112	0.821	89.108	93.115	98.443
113	0.826	89.234	93.236	98.533
114	0.831	89.391	93.371	98.642
115	0.836	89.525	93.506	98.751
116	0.840	89.657	93.624	98.868
117	0.845	89.777	93.742	98.974
118	0.850	89.906	93.856	99.066
119	0.855	90.032	93.982	99.143
120	0.860	90.136	94.091	99.210

Data Point No.	p_{Hg} (MPa)	$V_{Hg} - U$ (mm^3g^{-1})	V_{Hg} (mm^3g^{-1})	$V_{Hg} + U$ (mm^3g^{-1})
121	0.865	90.251	94.224	99.283
122	0.870	90.360	94.325	99.351
123	0.875	90.467	94.452	99.416
124	0.880	90.576	94.537	99.484
125	0.884	90.703	94.639	99.569
126	0.889	90.815	94.727	99.653
127	0.894	90.916	94.810	99.732
128	0.899	91.016	94.880	99.805
129	0.904	91.111	94.972	99.849
130	0.909	91.202	95.041	99.887
131	0.914	91.276	95.143	99.934
132	0.919	91.345	95.222	99.992
133	0.924	91.413	95.322	100.070
134	0.928	91.487	95.388	100.140
135	0.933	91.562	95.486	100.210
136	0.938	91.647	95.539	100.270
137	0.943	91.723	95.615	100.330
138	0.948	91.808	95.671	100.380
139	0.953	91.868	95.751	100.420
140	0.958	91.934	95.800	100.480
141	0.963	91.988	95.883	100.530
142	0.968	92.062	95.939	100.590
143	0.973	92.121	96.005	100.640
144	0.977	92.185	96.088	100.670
145	0.982	92.261	96.154	100.720
146	0.987	92.333	96.215	100.770
147	0.992	92.400	96.261	100.810
148	0.997	92.474	96.314	100.820
149	1.002	92.531	96.357	100.840
150	1.007	92.607	96.405	100.910
151	1.012	92.661	96.441	100.970
152	1.017	92.710	96.499	101.030
153	1.021	92.753	96.557	101.090
154	1.026	92.801	96.604	101.160
155	1.031	92.861	96.664	101.210
156	1.036	92.905	96.711	101.230
157	1.041	92.942	96.762	101.240
158	1.046	92.992	96.812	101.250
159	1.051	93.031	96.850	101.280
160	1.056	93.074	96.901	101.310

Annex

Discrete values of the reference curve with simultaneous confidence 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	93.125	96.941	101.330
162	1.065	93.168	96.979	101.350
163	1.070	93.213	97.025	101.370
164	1.075	93.259	97.060	101.410
165	1.080	93.303	97.099	101.440
166	1.085	93.356	97.129	101.480
167	1.090	93.392	97.173	101.500
168	1.095	93.423	97.196	101.520
169	1.100	93.454	97.221	101.550
170	1.105	93.488	97.254	101.590
171	1.109	93.521	97.274	101.620
172	1.114	93.557	97.333	101.650
173	1.119	93.589	97.371	101.660
174	1.124	93.607	97.447	101.660
175	1.129	93.625	97.484	101.660
176	1.134	93.657	97.539	101.680
177	1.139	93.695	97.585	101.700
178	1.144	93.739	97.623	101.720
179	1.149	93.779	97.639	101.740
180	1.153	93.826	97.656	101.770
181	1.158	93.891	97.667	101.800
182	1.163	93.933	97.686	101.830
183	1.168	93.976	97.698	101.860
184	1.173	94.005	97.726	101.890
185	1.178	94.036	97.755	101.910
186	1.183	94.058	97.782	101.940
187	1.188	94.079	97.811	101.950
188	1.192	94.095	97.840	101.950
189	1.197	94.110	97.860	101.980
190	1.202	94.117	97.878	102.000
191	1.207	94.136	97.897	102.030
192	1.212	94.169	97.923	102.040
193	1.217	94.207	97.952	102.060
194	1.222	94.243	97.979	102.080
195	1.227	94.274	98.007	102.110
196	1.232	94.288	98.034	102.110

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	94.295	98.063	102.110
198	1.241	94.303	98.076	102.120
199	1.246	94.322	98.085	102.130
200	1.251	94.357	98.089	102.150
201	1.256	94.390	98.101	102.160
202	1.261	94.417	98.115	102.190
203	1.266	94.437	98.124	102.210
204	1.271	94.463	98.142	102.210
205	1.276	94.492	98.154	102.210
206	1.281	94.504	98.175	102.210
207	1.285	94.518	98.204	102.210
208	1.290	94.525	98.229	102.210
209	1.295	94.538	98.251	102.210
210	1.300	94.549	98.286	102.210
211	1.305	94.557	98.311	102.210
212	1.310	94.571	98.336	102.210
213	1.315	94.577	98.347	102.210
214	1.320	94.593	98.358	102.210
215	1.325	94.618	98.363	102.210
216	1.329	94.643	98.374	102.210
217	1.334	94.664	98.391	102.210
218	1.339	94.701	98.412	102.210
219	1.344	94.729	98.428	102.210
220	1.349	94.761	98.450	102.210
221	1.354	94.768	98.464	102.210
222	1.359	94.774	98.476	102.210
223	1.364	94.780	98.489	102.210
224	1.369	94.789	98.494	102.210
225	1.373	94.803	98.505	102.210
226	1.378	94.811	98.512	102.210
227	1.383	94.823	98.525	102.210
228	1.388	94.852	98.537	102.210
229	1.393	94.869	98.549	102.210
230	1.398	94.876	98.562	102.210
231	1.403	94.876	98.574	102.210
232	1.408	94.883	98.574	102.210

V_{Hg} Certified pressure-volume curve (reference curve)

$V_{Hg} - U$ Lower limit curve of confidence band at significance level 0.95

$V_{Hg} + U$ Upper limit curve of confidence band at significance level 0.95