National Institute of Standards & Technology

Certificate

Standard Reference Material® 1019b

Glass Beads - Particle Size Distribution

This Standard Reference Material (SRM) is intended primarily for use in evaluating and calibrating particle size measurement instrumentation covering the 750 μm to 2 450 μm range. It consists of a single bottle containing approximately 200 g of solid spherical soda-lime glass beads. Typical use is in the evaluation of wire cloth test sieves in the range from No. 20 (850 μm) through No. 10 (2 000 μm). This size range follows that of the finer beads of SRM 1018b.

The certified cumulative volume (mass) distribution was determined using both calibrated scanning electron microscopy (SEM) and standard sieving procedures on samples chosen using a stratified random selection process. The certified values are the average of results from SEM analyses on five bottles. The sieve analyses of ten bottles were used to determine the variability between bottles as well as for a comparison with the SEM results.

Expiration of Certification: The certification of this SRM is valid indefinitely within the measurement uncertainties specified, provided the SRM is used in accordance with the instructions given in this certificate. However, it is expected that some beads will be lost with each use. When the unit's loss exceeds 2 % of the original mass, or if spillage or contamination occurs, the certification will be nullified and use of the SRM unit should be discontinued.

SEM Certification Procedure: Sample preparation for the SEM involved both a reduction in mass and a separation into size fractions. This was to achieve a representative sampling of the different size fractions, and a balanced statistical measure of each size fraction. The five test bottles were sieved into nine size fractions and then riffle split with a spinning microriffler. Backscatter electron images were taken at five different magnifications to obtain both adequate counting statistics and diameter resolution for particles in each size range. These 1 024 by 1 024 pixel images of the beads were acquired from the SEM into a computer as grey scale image files via a digital interface. Image analysis software was used to obtain the major and minor diameters of each glass bead based on the assumption of ellipsoidal particle shape. Diameters (in pixels) were converted to particle volume (prolate spheroid) and particle diameter (mean of major and minor diameters) using a micrometer slide calibrated at NIST.

Approximately 2 000 beads from each bottle were measured by SEM. Particle size distributions describing the percentage of mass represented by beads with diameters less than a given length were calculated using the weighting factors obtained from the sieving results. The SEM results for cumulative mass distribution of the five samples are shown in Figure 1. Table I is a listing of certified bead diameter values versus cumulative mass fraction. In that table, each mass fraction value is considered exact with uncertainty associated with the diameter value. At each mass fraction, the certified diameter and the expanded uncertainty define a 95 % prediction interval. Expanded uncertainties computed according to the ISO and NIST Guides [1] include allowances for measurement imprecision and material variability. The 95 % prediction interval at each mass fraction predicts where the true diameter lies for 95 % of the bottles of this SRM. Additionally, Table II presents the variables reversed with diameters sequenced as exact values from 760 μm to 2 460 μm, and the uncertainties associated with the certified mass fractions.

The technical direction, SEM measurements, sieve analysis, and statistical analysis leading to the certification were provided by J.F. Kelly of the NIST Ceramics Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by R.J. Gettings.

Gaithersburg, MD 20899
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Thomas E. Gills, Chief
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Statistical review was performed by K.R. Eberhardt of the NIST Statistical Engineering Division.

Sieve Analysis Procedure: The sieve testing was designed to provide reference values for sieve analysis as well as measure the between bottle variability (homogeneity). Ten bottles were selected from thirty-six bottles using a stratified random sampling plan. The results in Table II are from a series of sieve analyses performed following recommendations in ASTM SP 447B [2]. A stacked set of seven 200 mm (8 in) diameter sieves plus pan was shaken in a sieving unit for a 15 min vibration time. Ten bottles were sieved with an average material loss of 0.1 g from a 200 g bottle. The effective diameters were obtained by comparing the mass percentage of glass beads passing through a sieve with the certified diameter for that percentage as listed in Table I. Each of the effective diameters is well within the ASTM Specification [3] for permissible variation of average opening from the nominal sieve opening.

Each of the ten bottles was sieved twice according to randomized run order. This repetition measures reproducibility of the technique and assesses bottle to bottle variation in the particle size distribution. The mass of beads retained on each sieve was used to calculate the mass percent finer than that sieve. This is the ratio of the mass of beads passing through a sieve to the total starting mass. The results of replicate sieving for each bottle (Runs "1" and "2" are given in Table III as mass percent of beads passing through each successive screen. A graphical comparison of the mean of the five distributions obtained by SEM analysis with the mean of the twenty sieve analysis distributions is shown in Figure 2. The diameter values for the sieve analyses were obtained by using the nominal ASTM mesh opening for each sieve.

Table IV shows a comparison of the nominal sieve openings with the effective sieve openings for the set of sieves used in this study. This was determined by matching the percentage of beads passing through each sieve with the SEM results in Table I. The corresponding diameter from Table I is then the effective sieve opening. For example, the average percentage passing the 16 mesh screen for all bottles tested was 49.59 %. Interpolation between the 49 % (1 205 μm) and 50 % (1 211 μm) values gives an effective opening of 1 209 μm. This compares with the nominal opening of 1 180 μm.

Instructions for Use: The entire bottle unit of beads should be used in any application of this SRM. If this is impractical, special care must be exercised when taking subsamples from the SRM bottle. The recommended procedure is to use a microriffler to divide the 200 g sample into subsamples until a suitable subsample mass is obtained.

Using Calibrated Glass Beads for the Evaluation of the Effective Opening of Test Sieves: The allowed variation in sieve openings makes it difficult to compare size determinations made with different sets of sieves even though each set complies with the applicable ASTM, ANSI, or ISO test standard. The aperture size of a sieve can be determined as the average size of the openings in the sieve. However, the purpose of a sieve is to measure the size of particles and therefore, it is the effective opening that must be determined. This effective opening is determined by the size of the calibrated glass beads that will just pass through the sieve. This in turn permits the measurement of the particle size of an unknown material that will also just pass through the sieve.

The openings of a sieve are not all the same size; particles that are coarser than the average opening can pass through the larger holes. In addition, the separation achieved by a sieve is not sharp. A few particles capable of passing the sieve are always retained. The number of particles retained or passed depends on the manner and time of shaking and any measurement of the effective opening must take these variables into account. To a large extent, the glass bead (sphere) method of calibration automatically includes these effects because the sieves are shaken in the same manner, when being calibrated, as when measuring an unknown material.

The sieve openings are essentially square; particles of irregular shape can pass through although one dimension of the particle is considerably larger than the size of the opening. The average dimension of irregular particles that pass a sieve cannot be considered equal to the effective opening of the sieve as measured by the diameter of spheres that just pass.

To evaluate the effective opening of standard 203 mm (8 in) or 305 mm (12 in) test sieves with this SRM, the entire bottle of beads should be poured onto the top sieve screen. The sieves are then shaken in the same manner as that to be followed in routine analysis. To prevent blinding of a screen, the beads should not be used with a single screen; it is recommended that two relief screens be used to reduce the mass of beads. A rough rule of thumb is keep the loading below six layers of beads. For use with 76 mm (3 in) test sieves, the mass of beads must be reduced with a spinning riffler.

After the shaking has been completed, the stack of sieves is disassembled, and the beads are removed from each sieve and placed into a suitable weighing bottle. To reduce bead loss during this step, the transfer operation should utilize a large funnel or be carried out over glazed paper to recover any spillage. A soft brush is useful in removing the beads from the sieve and funnel.

Each of the sieve fractions is weighed to a precision of at least 0.01 g. After weighing, all beads are returned to the original SRM bottle and kept for reuse. The mass percent retained on each sieve is used to calculate the mass percent finer as the ratio of the mass of beads passing through a sieve to the total starting mass. The effective size of the sieve opening is determined by interpolation between the nearest values given in Table I.

The above calibration procedure is for use in comparison of sieve results and as a method to periodically monitor for changes in screens after service. This procedure is **not** to be used as a certification for test sieves. For assistance in complying with the calibration of wire cloth sieves according to ASTM E-11 specifications, contact the NIST Calibration Program at (301) 975-3471 or (301) 975-2002.

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9 1st Ed. ISO Geneva, Switzerland, (1993): see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994).

[2] "Manual on Test Sieving Methods," ASTM Special Technical Publication 447B, Philadelphia, PA, (1985).

[3] ASTM E 11-95, Standard Specification for Wire Cloth and Sieves for Testing Purposes, ASTM Annual Book of Standards, **Vol. 14.02**, West Conshohocken, PA, (1996).

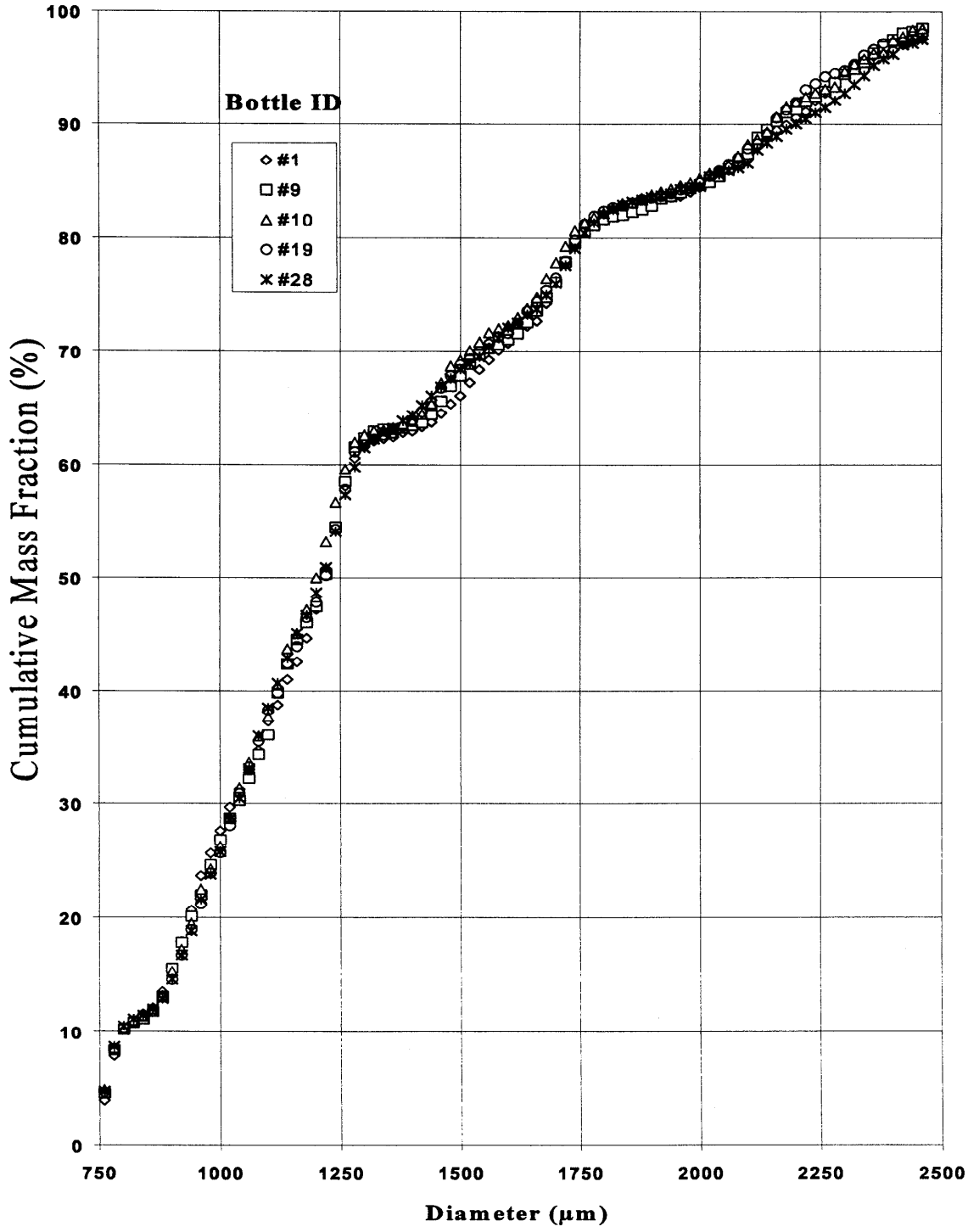

Figure 1. SEM Determination of Size Distribution for 5 Bottles of SRM 1019b

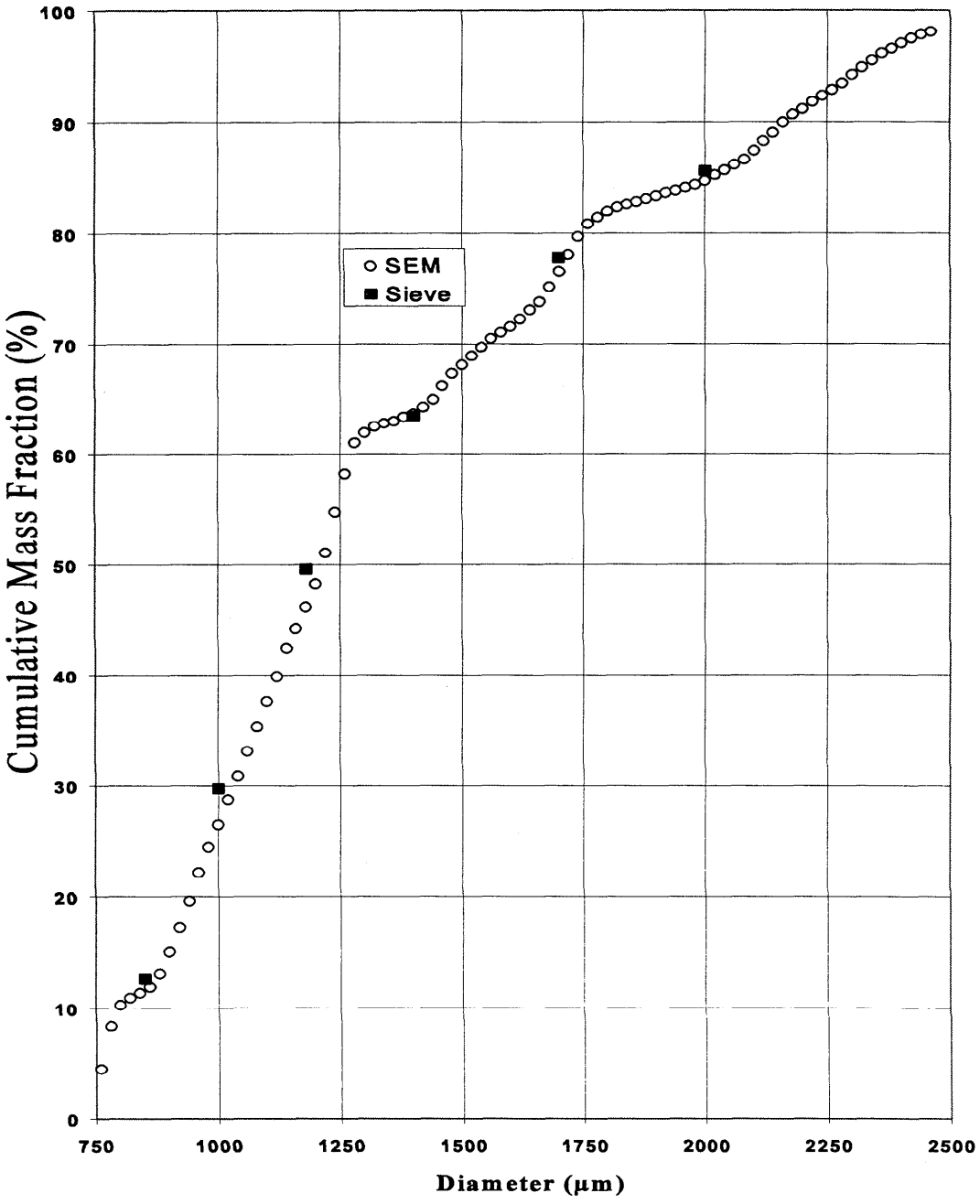

Figure 2. Comparison of SRM 1019b SEM and Sieve Data

Table I. Certified Bead Diameters (μm) Versus Mass Fraction (%)

Mass (%)	Diameter (μm)	Uncertainty* ± (μm)	Mass (%)	Diameter (μm)	Uncertainty* ± (μm)	Mass (%)	Diameter (μm)	Uncertainty* ± (μm)
2	746	15	35	1079	21	68	1496	36
3	753	14	36	1085	22	69	1517	39
4	758	14	37	1094	22	70	1547	39
5	763	14	38	1104	22	71	1579	40
6	767	14	39	1112	22	72	1609	40
7	773	14	40	1120	23	73	1640	36
8	778	15	41	1130	22	74	1659	34
9	784	15	42	1136	23	75	1676	33
10	797	18	43	1147	24	76	1692	33
11	824	24	44	1158	24	77	1704	33
12	861	22	45	1167	24	78	1717	33
13	878	18	46	1178	23	79	1729	33
14	889	17	47	1188	24	80	1743	34
15	899	18	48	1199	24	81	1765	38
16	908	18	49	1205	23	82	1804	47
17	918	18	50	1211	23	83	1865	58
18	927	18	51	1218	23	84	1950	57
19	934	19	52	1225	23	85	2009	48
20	942	19	53	1230	24	86	2059	44
21	950	19	54	1235	23	87	2090	43
22	959	19	55	1240	24	88	2115	43
23	967	19	56	1247	24	89	2140	44
24	976	19	57	1252	24	90	2166	47
25	984	20	58	1259	24	91	2195	52
26	996	20	59	1266	24	92	2228	53
27	1004	20	60	1272	25	93	2260	52
28	1012	20	61	1282	27	94	2290	52
29	1022	20	62	1301	33	95	2320	50
30	1031	20	63	1354	51	96	2358	51
31	1041	20	64	1416	40	97	2398	53
32	1050	20	65	1440	35	98	2449	59
33	1059	20	66	1459	35			
34	1069	21	67	1477	37			

*The uncertainty at each percentile, computed according to the ISO Guide [1], is an expanded uncertainty at the 95 % level of confidence which includes uncertainty due to measurement imprecision, SEM calibration, and material variability. Each certified diameter with its expanded uncertainty define a diameter range within which the true diameter is expected to lie for at least 95 % of the bottles of this SRM.

Table II. Certified Mass Fractions (%) Versus Bead Diameter (μm)

Diameter (μm)	Mass (%)	Uncertainty* ± (%)	Diameter (μm)	Mass (%)	Uncertainty* ± (%)	Diameter (μm)	Mass (%)	Uncertainty* ± (%)
760	4.5	2.5	1340	62.8	1.0	1920	83.7	1.0
780	8.4	2.2	1360	63.0	1.0	1940	83.9	1.1
800	10.3	1.5	1380	63.4	1.1	1960	84.1	1.1
820	10.9	1.0	1400	63.7	1.3	1980	84.4	1.3
840	11.4	1.0	1420	64.3	1.6	2000	84.7	1.4
860	11.9	1.2	1440	65.0	1.8	2020	85.3	1.4
880	13.1	1.7	1460	66.2	2.0	2040	85.7	1.4
900	15.1	2.0	1480	67.3	2.0	2060	86.2	1.5
920	17.3	2.1	1500	68.1	2.0	2080	86.6	1.7
940	19.6	2.2	1520	68.9	1.8	2100	87.4	1.8
960	22.2	2.2	1540	69.7	1.7	2120	88.3	1.9
980	24.5	2.2	1560	70.5	1.7	2140	89.1	1.9
1000	26.5	2.2	1580	71.1	1.5	2160	90.0	1.9
1020	28.8	2.1	1600	71.6	1.6	2180	90.7	1.9
1040	30.9	2.1	1620	72.3	1.6	2200	91.2	1.8
1060	33.1	2.2	1640	73.1	1.7	2220	91.8	1.8
1080	35.3	2.3	1660	73.8	2.0	2240	92.4	1.8
1100	37.6	2.4	1680	75.2	2.2	2260	92.9	1.9
1120	39.9	2.4	1700	76.6	2.3	2280	93.5	1.9
1140	42.5	2.3	1720	78.1	2.4	2300	94.2	1.9
1160	44.3	2.2	1740	79.7	2.1	2320	94.9	1.8
1180	46.2	2.3	1760	80.9	1.8	2340	95.5	1.7
1200	48.3	2.6	1780	81.5	1.5	2360	96.1	1.7
1220	51.1	3.0	1800	82.0	1.4	2380	96.5	1.6
1240	54.8	3.0	1820	82.4	1.2	2400	97.0	1.5
1260	58.2	2.8	1840	82.7	1.1	2420	97.5	1.5
1280	61.0	2.3	1860	82.9	1.1	2440	97.8	1.4
1300	62.0	1.5	1880	83.1	1.1	2460	98.0	1.4
1320	62.5	1.2	1900	83.4	1.1			

*The uncertainty at each percentile, computed according to the ISO Guide [1], is an expanded uncertainty at the 95 % level of confidence which includes uncertainty due to measurement imprecision, SEM calibration, and material variability. Each certified mass fraction with its expanded uncertainty define a percentage range within which the true mass fraction is expected to lie for at least 95 % of the bottles of this SRM.

Table III. Mass Fraction Passing Each Sieve

Run 1					Bottle #					
Sieve (No.)	1	9	10	14	18	19	23	27	28	32
					Mass Fraction (%)					
8	97.87	97.78	98.13	98.05	97.79	97.86	98.04	98.15	97.91	97.97
10	85.37	85.21	85.80	85.47	85.55	85.69	85.73	85.65	85.80	85.93
12	77.80	77.61	77.87	77.49	77.79	77.62	77.93	77.65	77.86	78.34
14	62.98	63.45	63.61	63.29	63.35	63.19	63.38	63.45	63.58	63.99
16	49.17	49.53	49.89	49.65	49.65	49.55	49.87	49.63	49.93	50.04
18	29.47	29.54	29.97	29.91	29.75	29.60	30.02	29.80	29.96	30.11
20	12.55	12.52	12.76	12.71	12.66	12.65	12.84	12.63	12.72	12.86
25	1.75	1.69	1.79	1.77	1.78	1.80	1.78	1.74	1.79	1.76
Run 2										Average
8	97.86	97.80	98.16	98.07	97.83	97.89	98.02	98.11	97.92	97.95	97.96
10	85.32	85.21	85.92	85.49	85.78	85.59	85.74	85.69	85.82	85.86	85.63
12	77.85	77.54	78.43	77.52	77.85	77.66	77.93	77.62	77.77	78.02	77.81
14	62.98	63.43	63.64	63.28	63.35	63.33	63.35	63.44	63.55	63.92	63.43
16	48.86	49.54	49.87	49.35	49.44	49.33	49.61	49.56	49.40	49.87	49.59
18	29.21	29.44	29.70	29.67	29.65	29.65	29.93	29.86	29.75	29.91	29.74
20	12.48	12.53	12.65	12.62	12.64	12.59	12.83	12.58	12.62	12.80	12.66
25	1.60	1.68	1.67	1.64	1.67	1.71	1.72	1.71	1.69	1.66	1.72

Table IV. Comparison of Nominal and Effective Sieve Openings

Sieve (No.)	Sieve Opening (μm)	
	Nominal	Effective
10	2000	2040
12	1700	1715
14	1400	1381
16	1180	1209
18	1000	1028
20	850	872

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax: (301) 926-4751, e-mail: srminfo@nist.gov, or WWW: http://ts.nist.gov/srm.