National Institute of Standards & Technology

Certificate

Standard Reference Material® 1004b

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 40 µm to 150 µm range. SRM 1004b is one of a series of SRMs for particle size analysis and lies between that of the finer beads of SRM 1003b and the coarser beads of SRM 1017b. SRM 1004b is commonly used in the evaluation of test sieves in the range from No. 270 (53 µm) through No. 120 (125 µm). A unit of SRM 1004b consists of a single bottle containing approximately 43 g of solid spherical borosilicate glass beads.

Certified Values: A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [1]. The certified cumulative volume (or 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.

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 seven size fractions and then riffle split with a spinning microriffler. Backscatter electron images were taken at six different magnifications to obtain both adequate counting statistics and diameter resolution for particles in each size range. Figure 1 is an example of one of the sieve fraction images. These 2048 by 2048 pixel images of the beads were acquired from the SEM into a computer as greyscale 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 a particle volume (prolate spheroid) and particle diameter (geometric mean of major and minor diameters) using a micrometer slide calibrated at NIST.

Expiration of Certification: The certification of **SRM 1004b** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified. It is expected that some spheres will be lost with each use. If 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 should be discontinued.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

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.

Statistical consultation was provided by K.R. Eberhardt of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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Certificate Revision History on Last Page

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Approximately 4000 beads were measured per bottle. Particle size distributions describing the percentage of powder volume represented by particles 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 sample bottles are shown in Figure 2. Table 1 is a listing of certified bead diameter values versus cumulative mass fraction with the mass fraction sequenced from 1 % to 99 % in 1 % increments. In that table, each mass fraction value is considered exact with its 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 2 presents the variables reversed with diameters sequenced as exact values from 40 µm to 146 µm and the uncertainties associated with the certified mass fractions.

Sieve Analysis Procedure: The sieve testing was designed to provide a measure of the between-bottle variability (homogeneity) and a check for the certified values. Ten bottles were selected from the total population of bottles using a stratified random sampling plan. Each of the ten bottles was sieved twice with a randomized run order. This repetition measures repeatability of the technique and assesses bottle-to-bottle variation in the particle size distribution. Mechanical sieving, using a Tyler Ro-Tap[1], was done following recommendations in ASTM STP 447 [2]. The entire bottle of beads was poured onto the top sieve and the sieves were then shaken in the Ro-Tap for 15 min. After the shaking was completed, the stack of sieves was disassembled, and the beads removed from each sieve and weighed to a precision of 0.01 g. After weighing, all beads were returned to the original container and reused in Run 2. 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. For illustration purposes, the results of replicate sieving for each bottle (Runs "1" and "2") are given in Table 3 as mass percent of beads passing through each successive sieve 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 3. The diameter values for the sieve analyses were obtained by using the nominal ASTM mesh opening for each sieve.

Table 4 shows an example comparison of the nominal sieve opening with the effective sieve size opening for sieves used at NIST. This was determined by matching the mass fraction of beads passing through each sieve with the values in Table 1. The corresponding diameter from Table 1 is then the effective sieve opening. For example, the average percentage passing the 200 mesh sieve screen for all bottles tested was 46.2 %. Interpolation between the 46 % (74.6 µm) and 47 % (75.6 µm) values gives an effective opening of 74.8 µm. This compares with the nominal opening of 75 µm. Each of the effective diameters is well within the ASTM Specification [3] for permissible variation of average opening from the nominal sieve opening.

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 43 g sample into subsamples until a suitable subsample mass is obtained. Before and after the sieving procedure, weigh the sample mass to determine the mass of beads lost.

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 screen 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 spheres 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, and 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. Recognizing that the number of particles retained or passed depends on the manner and time of shaking, any measurement of the effective opening must take these variables into account. To a large extent, the glass 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.

[1] Certain commercial equipment, instruments, or materials are identified to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

The sieve openings are essentially square allowing particles of irregular shape to pass through although one dimension of the particle is considerably larger than the size of the opening. Therefore, the average dimension of irregular particles that pass a sieve cannot be considered equal to the effective opening of the sieve as determined using SRM 1004b.

To evaluate the effective opening of standard 203 mm (8 in) or 305 mm (12 in) diameter 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 used in routine analysis. To prevent blinding (overloading) of a screen, the beads should not be used with a single screen; typically two relief screens are needed to reduce the mass of beads. An individual screen's loading should be below six layers of beads at any given time (for use with 76 mm (3 in) test sieves, the mass of beads should be reduced with a spinning riffler).

After shaking, the stack of sieves is disassembled, and the beads are removed from each sieve and placed into a suitable weighing container. To reduce loss of material 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 1.

The above calibration procedure is for use in comparison testing of sieve results and as a method to systematically monitor for changes in sieve screens after service. NIST calibrations of wire cloth sieves according to ASTM E 11 specifications are available through the NIST Calibration Program at (301) 975-2200.

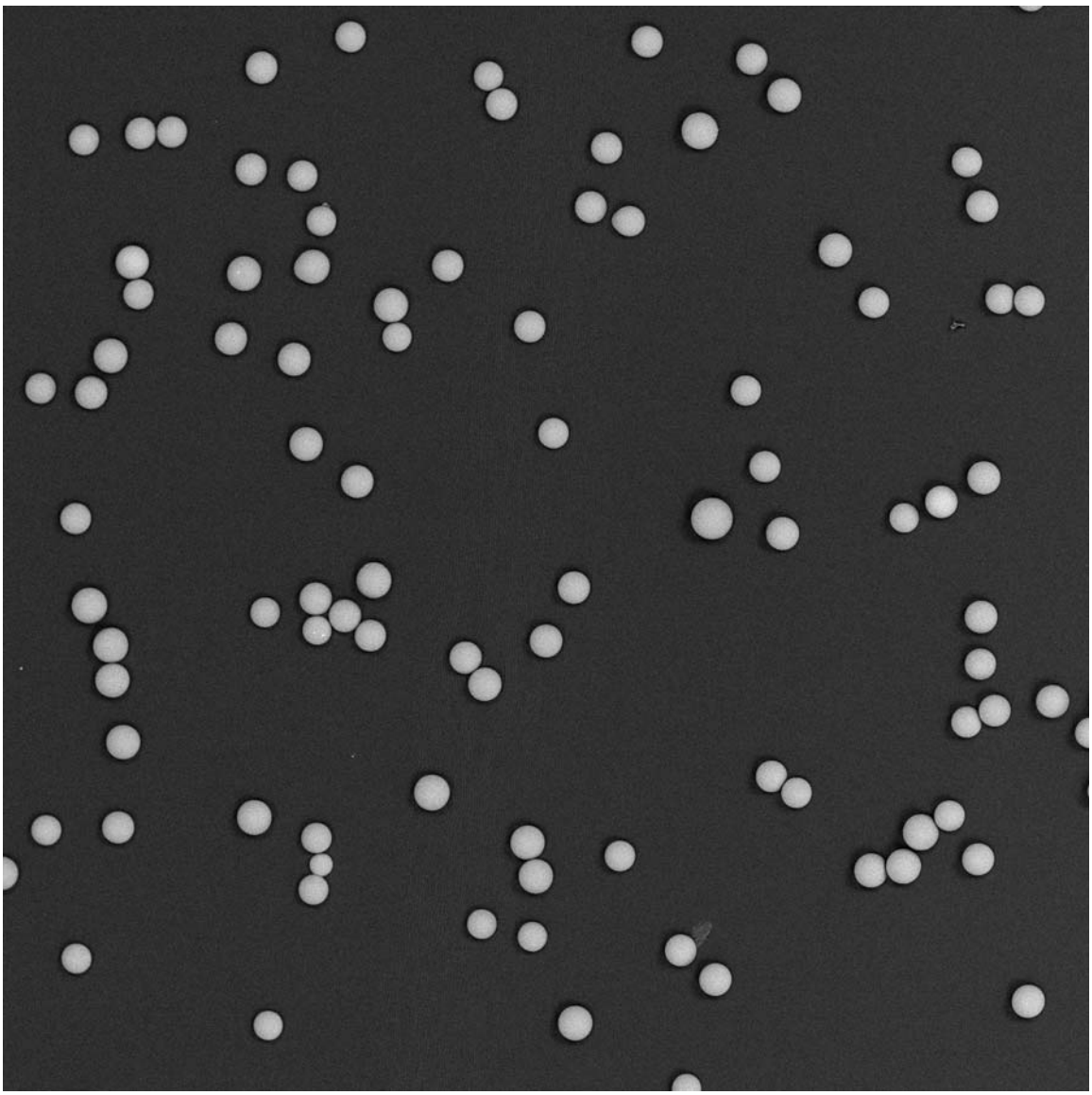

Figure 1. SEM Image of Glass Spheres

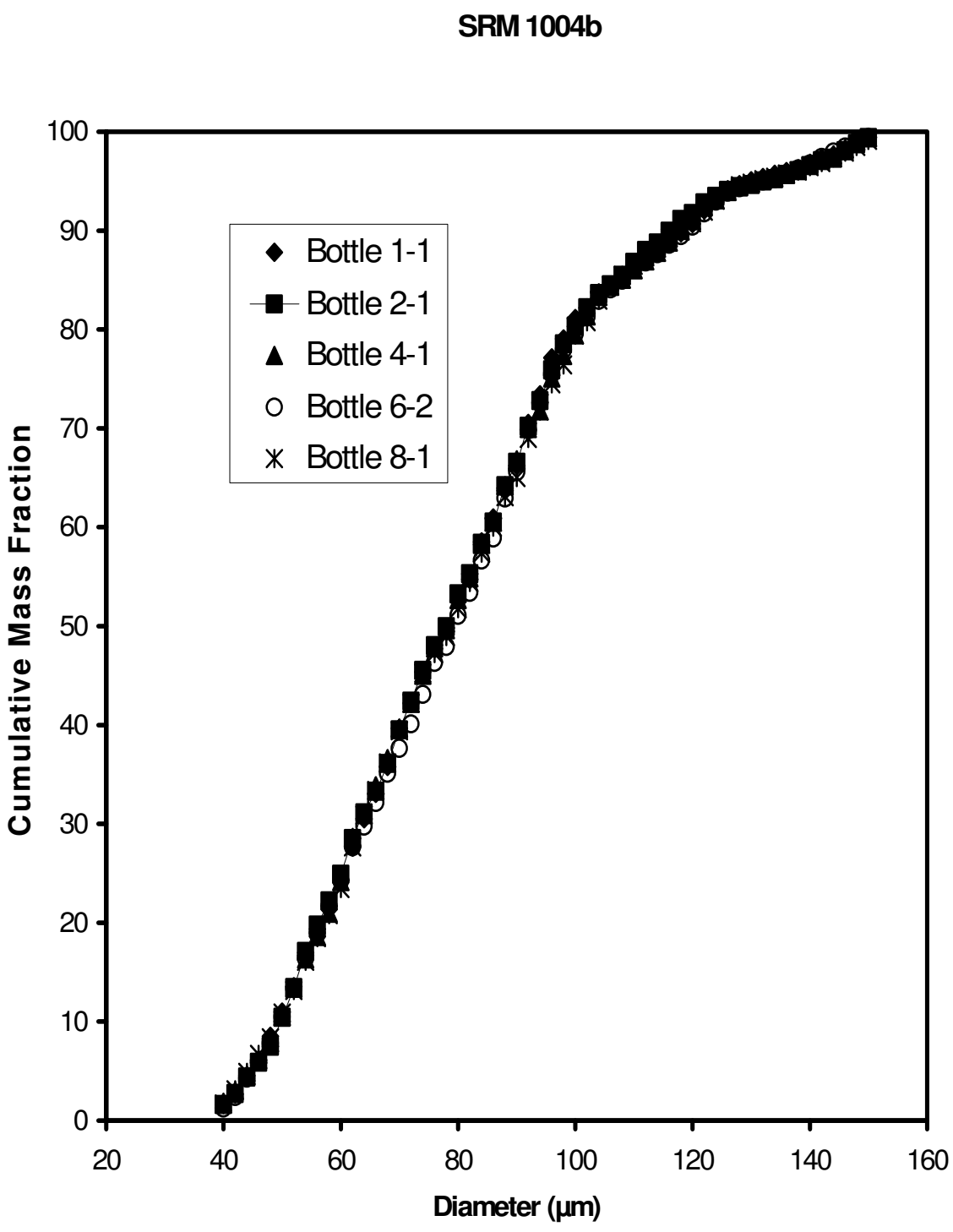

Figure 2. SEM Determination of Size Distribution for 5 Bottles of SRM 1004b

Table 1. Certified Diameters (µm) Versus Mass Fraction (%)

Mass (%)	Diameter (µm)	Uncertainty (±µm)	Mass (%)	Diameter (µm)	Uncertainty (±µm)	Mass (%)	Diameter (µm)	Uncertainty (±µm)
1	38.5	1.1	34	66.8	1.3	67	90.3	1.7
2	40.7	1.0	35	67.5	1.3	68	90.9	1.7
3	42.2	1.0	36	68.1	1.3	69	91.5	1.7
4	43.6	1.0	37	68.8	1.3	70	92.1	1.7
5	44.8	0.9	38	69.3	1.4	71	92.8	1.7
6	45.9	1.0	39	70.0	1.4	72	93.5	1.8
7	47.0	1.0	40	70.8	1.4	73	94.2	1.8
8	48.0	1.0	41	71.3	1.4	74	94.8	1.8
9	48.8	1.0	42	72.0	1.4	75	95.7	1.9
10	49.5	1.0	43	72.5	1.5	76	96.4	1.8
11	50.2	0.9	44	73.1	1.4	77	97.1	1.9
12	50.9	1.0	45	73.8	1.4	78	98.0	2.0
13	51.6	1.0	46	74.6	1.5	79	98.9	2.0
14	52.1	1.0	47	75.6	1.5	80	100.2	2.0
15	52.8	1.0	48	76.7	1.6	81	101.2	2.0
16	53.6	1.0	49	77.5	1.5	82	102.3	2.0
17	54.4	1.1	50	78.4	1.5	83	103.6	2.0
18	55.4	1.1	51	79.1	1.5	84	105.2	2.0
19	56.2	1.1	52	79.8	1.5	85	107.5	2.1
20	57.0	1.1	53	80.6	1.6	86	109.6	2.1
21	57.7	1.1	54	81.3	1.5	87	111.9	2.2
22	58.3	1.1	55	82.1	1.5	88	113.8	2.3
23	59.0	1.1	56	82.6	1.6	89	115.9	2.4
24	59.7	1.1	57	83.5	1.6	90	117.9	2.5
25	60.2	1.1	58	84.1	1.6	91	119.7	2.6
26	60.9	1.1	59	84.9	1.6	92	121.7	2.6
27	61.5	1.2	60	85.5	1.7	93	123.6	2.6
28	62.1	1.2	61	86.3	1.6	94	125.9	2.6
29	62.7	1.2	62	86.8	1.6	95	131.2	2.9
30	63.5	1.2	63	87.6	1.6	96	137.4	3.0
31	64.4	1.2	64	88.3	1.6	97	141.6	3.1
32	65.0	1.2	65	89.1	1.7	98	145.2	3.3
33	65.9	1.3	66	89.7	1.6	99	148.4	3.3

Table 2. Certified Mass Fractions (%) Versus Diameter (µm)

Diameter (µm)	Mass (%)	Uncertainty (%)	Diameter (µm)	Mass (%)	Uncertainty (%)
40	1.6	0.8	94	72.5	2.1
42	2.8	0.8	96	75.6	2.1
44	4.5	0.9	98	77.9	2.1
46	6.2	1.0	100	80.0	1.9
48	8.0	1.1	102	81.6	1.8
50	10.7	1.1	104	83.3	1.7
52	13.4	1.2	106	84.4	1.6
54	16.5	1.3	108	85.2	1.5
56	18.9	1.4	110	86.4	1.4
58	21.4	1.5	112	87.2	1.3
60	24.2	1.6	114	88.0	1.2
62	28.0	1.7	116	89.1	1.2
64	30.6	1.7	118	90.1	1.1
66	33.2	1.8	120	90.9	1.0
68	36.0	1.9	122	92.2	0.9
70	39.2	2.0	124	93.2	0.8
72	41.8	2.1	126	94.0	0.7
74	44.8	2.2	128	94.5	0.6
76	47.4	2.1	130	94.8	0.6
78	49.2	2.2	132	95.1	0.6
80	52.3	2.2	134	95.4	0.6
82	54.5	2.2	136	95.8	0.6
84	57.8	2.2	138	96.2	0.6
86	60.2	2.2	140	96.7	0.7
88	63.6	2.1	142	97.2	0.8
90	66.1	2.1	144	97.6	0.9
92	69.9	2.1	146	98.2	1.1

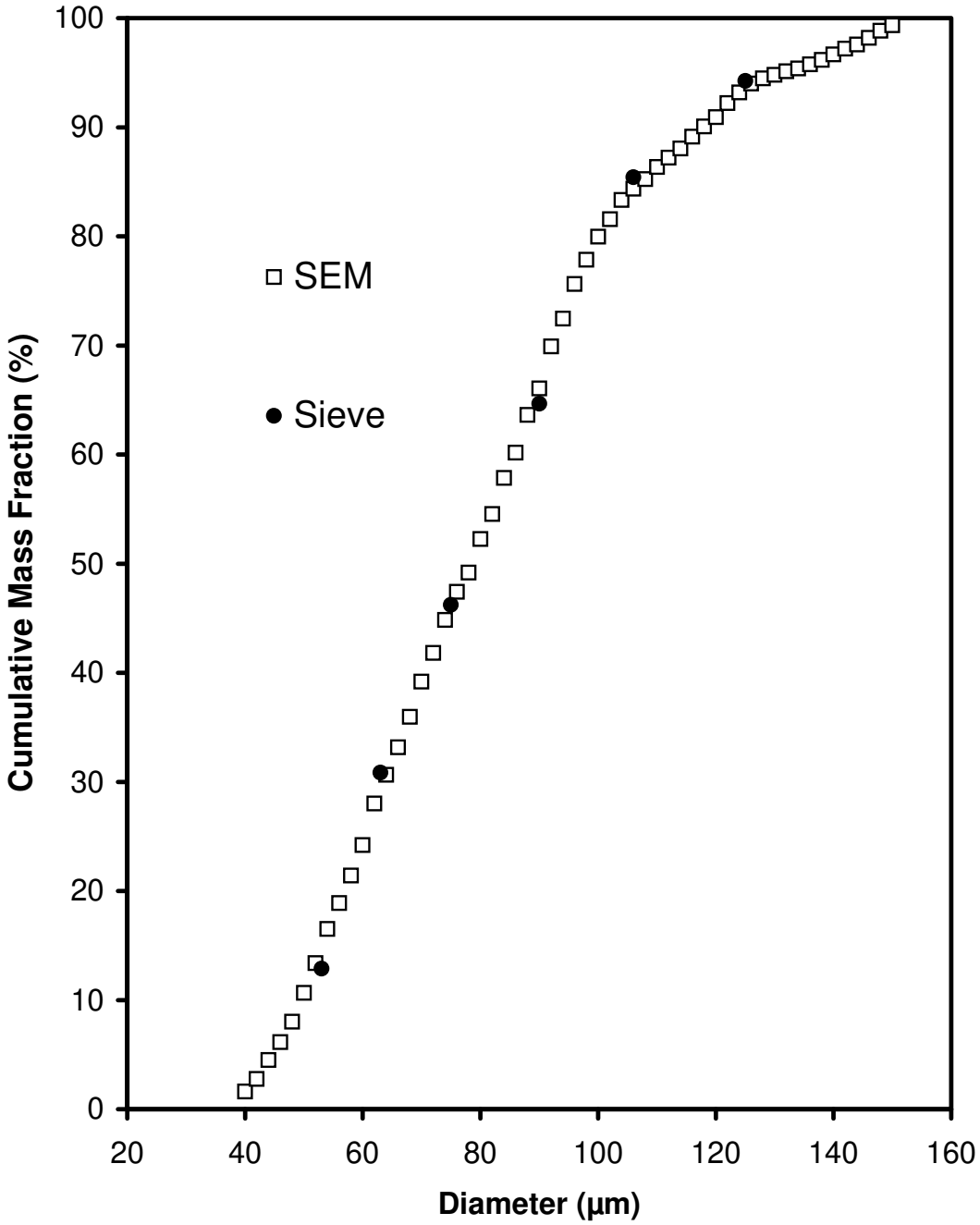

Figure 3. Comparison of SEM and Sieve Data for SRM 1004b.

Table 3. Mass Fraction Passing Each Sieve
Lot/Bottle Number

Sieve-Run	1/1	2/1	3/1	4/1	5/1	6/1	6/2	6/3	7/1	8/1	Mean
120-1	94.19	94.08	94.34	94.25	94.14	94.44	94.22	94.43	94.18	94.13	94.25
120-2	94.42	94.18	94.16	94.11	94.09	94.43	94.17	94.30	94.50	94.16	
140-1	85.34	85.22	85.59	85.42	85.35	85.72	85.43	85.66	85.44	85.39	85.43
140-2	85.45	85.31	85.42	85.21	85.25	85.78	85.38	85.39	85.47	85.37	
170-1	65.29	63.33	65.50	64.03	64.53	65.97	64.82	66.30	65.19	65.47	64.68
170-2	63.37	64.39	64.91	64.98	62.63	66.07	64.88	64.15	63.55	64.20	
200-1	46.47	45.91	46.52	46.30	45.66	46.74	46.37	46.83	46.21	46.63	46.22
200-2	45.51	45.93	46.62	46.00	46.34	46.76	46.17	45.68	45.66	46.11	
230-1	31.15	30.68	30.77	30.53	30.66	30.99	30.96	31.60	30.73	30.83	30.84
230-2	29.90	31.07	31.04	31.00	30.70	30.85	30.88	31.01	30.47	31.07	
270-1	13.16	12.71	12.72	13.08	12.56	13.57	13.41	13.15	13.34	12.99	12.88
270-2	12.50	12.82	12.71	12.68	12.42	12.93	13.08	12.47	12.44	12.79	

Table 4. Comparison of Nominal and Effective Sieve Openings

Sieve No.	Sieve Opening (μm)	
	Nominal	Effective
270	53	52
230	63	64
200	75	75
170	90	88
140	106	107
120	125	125

REFERENCES

[1] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Aug 2011); see also Taylor, B.N.; 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); available at http://www.nist.gov/physlab/pubs/index.cfm (accessed August 2011).

[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).

Certificate Revision History: 10 August 2011 (Change of certificate expiration, editorial changes); **29 September 2009** (Extension of certification period and editorial changes); **19 February 2008** (Extension of certification period and editorial changes); **02 March 2000** (Original certificate date)

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-2200; Fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.