National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1018b

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 220 μm to 750 μm range. The SRM consists of a single bottle containing approximately 87 g of solid spherical soda-lime glass beads. Typical use is in the evaluation of wire-cloth test sieves in the range from No. 60 (250 μm) through No. 25 (710 μm). This size range is intermediate between the finer beads of SRM 1017b and the coarser beads of SRM 1019b.

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 entailed 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 eight size fractions and then riffle split with a spinning microriffler to obtain a sample amount suitable for analysis by SEM. 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 1024 by 1024 pixel images of the particles 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. These diameters were converted to a particle volume (prolate spheroid) and particle diameter (mean of major and minor diameters). The pixel to length conversion was determined using a micrometer slide calibrated at NIST using laser interferometry.

Several hundred particles were measured by SEM for each sieve fraction for a total of approximately 3 000 beads measured per bottle. 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 with the mass fraction sequenced from 1 % to 99 % in 1 % increments. The 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 predicts where the true diameter lies for 95 % of the samples of this SRM. Additionally, Table II presents the variables reversed with diameters sequenced as exact values from 220 μm to 750 μm, and the uncertainty associated with the certified mass fraction.

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
Certificate Issue Date: April 22, 1997

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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 review was performed by L.M. Gill of the NIST Statistical Engineering Division.

Sieve Analysis Procedure: The sieve testing was designed to provide reference values for sieve analysis as well as a measure of 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 sieves (8 in) plus pan were shaken in a sieving unit for a 15 min vibration time. Ten bottles were sieved with an average material loss of 0.03 g from an 87 g bottle. The effective diameters are 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 with a 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 is 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 30 mesh screen for all bottles tested was 80.8 %. Interpolation between the 80 % (589.6 μm) and 81 % (595.6 μm) values gives an effective opening of 594 μm. This compares with the nominal opening of 600 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 87 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 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. 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 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 and 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 particles. A rough rule of thumb is keep the loading below six layers of particles. 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 loss of material during this step, the transfer should be done using a large funnel or 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. The compliance of wire cloth sieves according to ASTM E-11 specifications can be tested by contacting 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).

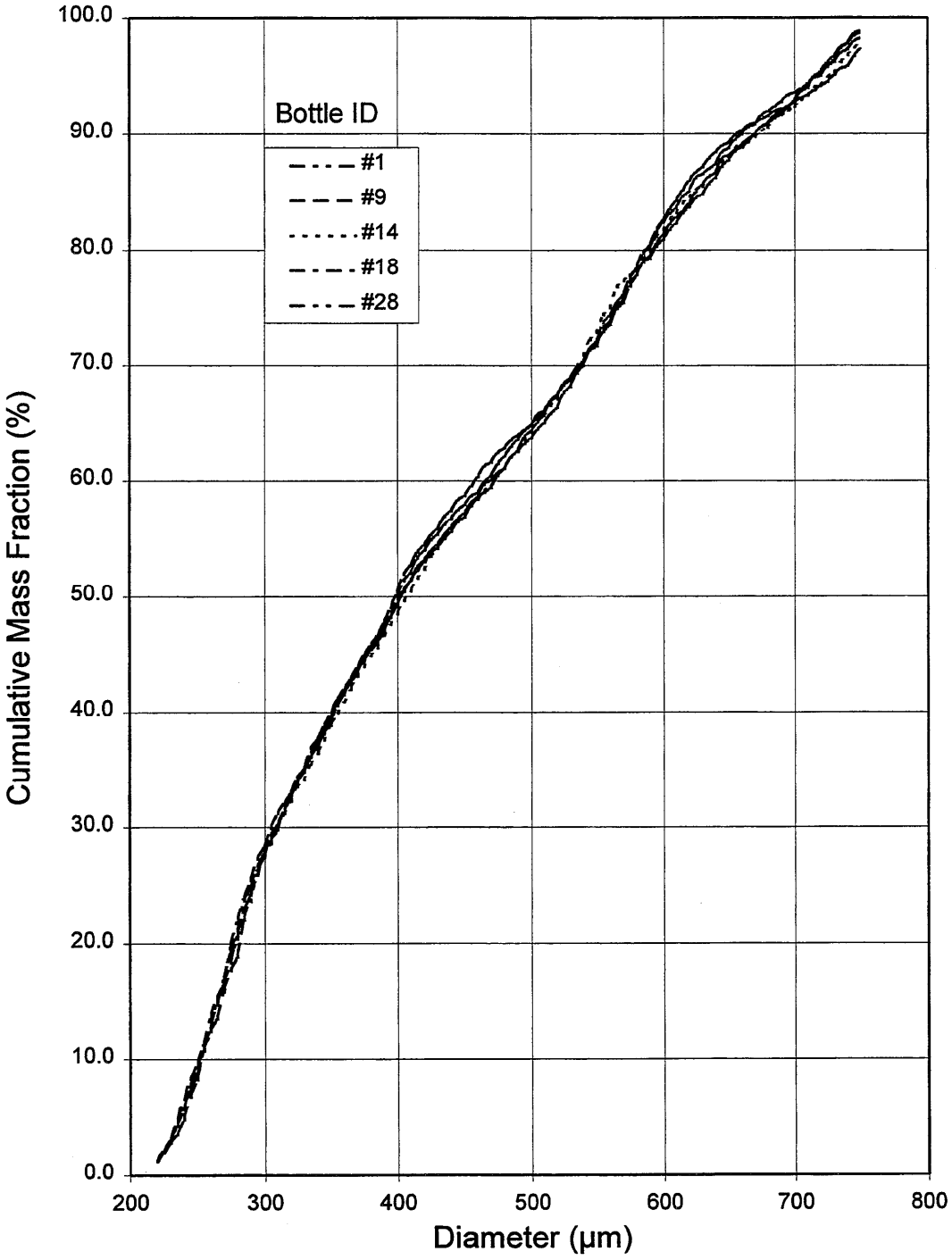

Figure 1. SEM Determination of Size Distribution for 5 Bottles of SRM 1018b

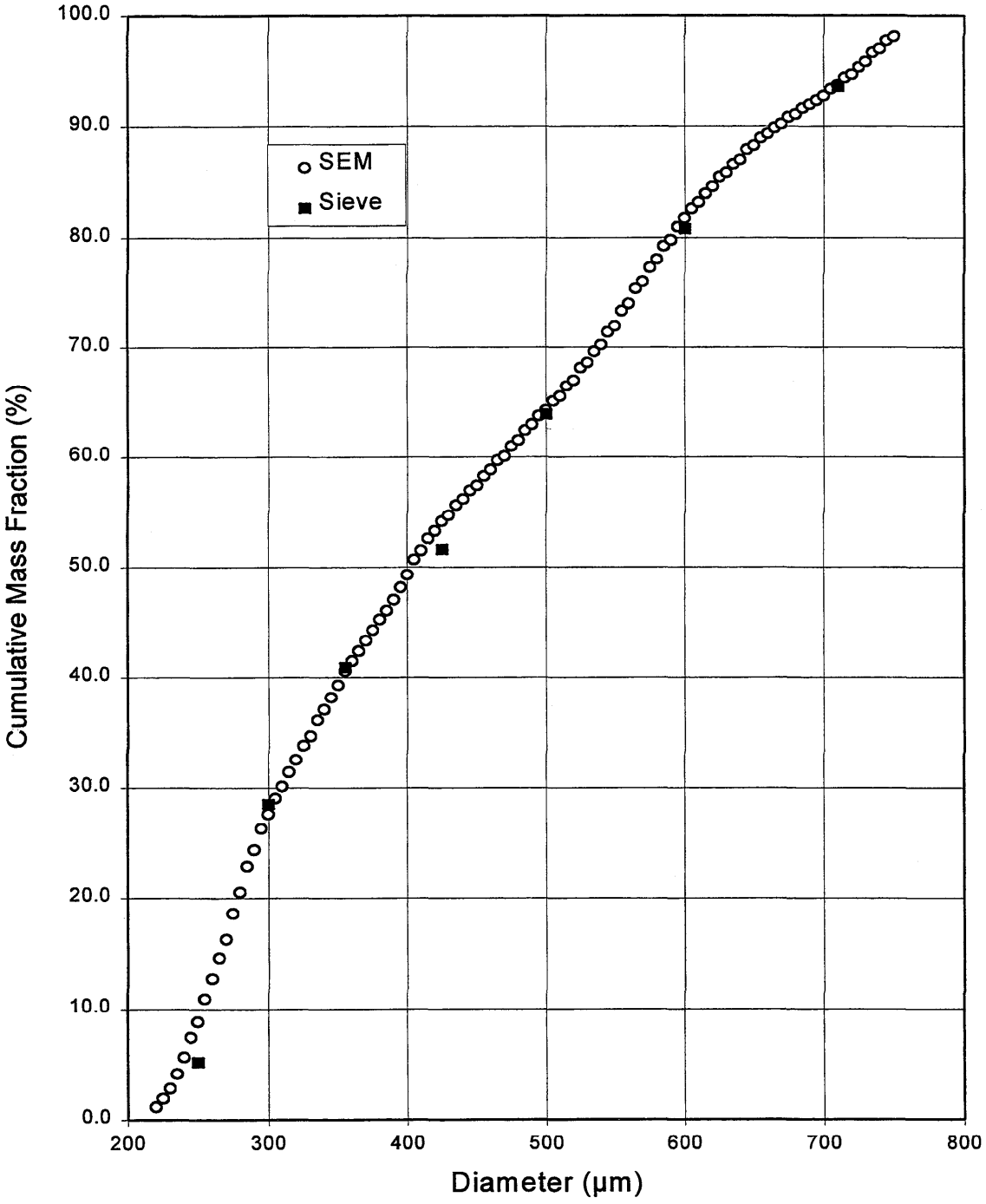

Figure 2. Comparison of SRM 1018b SEM and Sieve Data

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

Mass (%)	Diameter (μm)	Uncertainty* ± (μm)	Mass (%)	Diameter (μm)	Uncertainty* ± (μm)	Mass (%)	Diameter (μm)	Uncertainty* ± (μm)
1	218.1	3.1	34	326.0	4.3	67	518.6	6.9
2	226.1	3.0	35	330.8	4.5	68	524.3	6.7
3	231.6	3.0	36	335.0	4.6	69	530.9	6.8
4	234.6	3.4	37	339.4	4.8	70	537.6	6.9
5	238.1	3.4	38	343.8	4.6	71	542.9	7.0
6	240.7	3.4	39	347.6	4.7	72	548.9	7.5
7	243.7	3.4	40	352.3	4.8	73	554.3	7.4
8	247.0	3.5	41	356.5	5.0	74	558.3	7.6
9	250.1	3.4	42	362.0	5.0	75	564.3	7.6
10	252.3	3.4	43	367.5	4.9	76	568.3	7.7
11	255.7	3.4	44	372.5	5.0	77	573.0	8.0
12	258.0	3.5	45	377.5	5.0	78	579.0	7.5
13	261.1	3.6	46	383.5	5.6	79	584.3	7.8
14	263.3	3.7	47	388.5	5.3	80	589.6	7.9
15	266.2	3.7	48	393.0	5.6	81	595.6	8.0
16	268.5	3.6	49	397.9	5.7	82	601.0	8.6
17	271.7	3.5	50	401.9	6.1	83	607.6	8.7
18	274.0	3.6	51	406.1	5.9	84	615.0	9.7
19	275.7	4.0	52	411.6	6.7	85	622.8	10.0
20	278.6	4.0	53	417.0	7.3	86	629.3	10.4
21	281.4	4.1	54	423.7	7.1	87	638.3	10.4
22	283.1	4.2	55	430.9	7.6	88	646.3	10.2
23	285.8	4.0	56	437.5	7.6	89	655.4	10.2
24	288.3	3.9	57	444.9	8.1	90	666.3	10.9
25	290.9	4.2	58	452.4	7.9	91	677.7	10.8
26	294.3	4.1	59	460.2	8.4	92	690.0	10.8
27	296.9	4.0	60	468.2	8.9	93	700.7	10.5
28	300.6	4.1	61	474.9	9.0	94	711.7	10.2
29	304.4	4.2	62	482.7	8.5	95	722.4	10.8
30	308.8	4.3	63	488.3	7.9	96	730.4	11.2
31	312.8	4.7	64	496.2	7.9	97	738.4	10.7
32	317.4	4.2	65	504.9	7.4	98	747.7	11.1
33	320.8	4.2	66	512.3	7.1	99	761.9	14.0

*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 samples.

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

Diameter (μm)	Mass (%)	Uncertainty* ± (%)	Diameter (μm)	Mass (%)	Uncertainty* ± (%)	Diameter (μm)	Mass (%)	Uncertainty* ± (%)
220	1.2	0.4	400	49.3	1.5	580	78.0	1.4
225	2.0	0.5	405	50.7	1.4	585	79.3	1.6
230	2.8	0.8	410	51.5	1.4	590	79.8	1.3
235	4.2	1.0	415	52.6	1.2	595	81.0	1.7
240	5.7	1.3	420	53.3	1.3	600	81.7	1.3
245	7.4	1.3	425	54.2	1.1	605	82.6	1.4
250	8.8	1.1	430	54.7	1.2	610	83.2	1.2
255	10.9	1.4	435	55.7	1.1	615	84.0	1.5
260	12.7	1.5	440	56.2	1.2	620	84.6	1.4
265	14.6	1.6	445	57.0	1.0	625	85.5	1.5
270	16.3	1.5	450	57.5	1.2	630	85.9	1.2
275	18.6	1.7	455	58.3	1.0	635	86.6	1.3
280	20.5	1.9	460	58.9	1.2	640	87.0	1.4
285	22.9	1.8	465	59.7	1.1	645	87.9	1.3
290	24.4	1.5	470	60.1	1.2	650	88.3	1.2
295	26.3	1.3	475	61.0	1.1	655	89.0	1.1
300	27.6	1.2	480	61.6	1.3	660	89.4	1.1
305	29.1	1.4	485	62.4	1.2	665	89.9	1.0
310	30.1	1.0	490	63.0	1.1	670	90.2	1.0
315	31.5	1.3	495	63.8	1.0	675	90.8	0.9
320	32.5	1.2	500	64.3	1.0	680	91.1	1.0
325	33.8	1.1	505	65.1	1.0	685	91.7	0.8
330	34.7	0.9	510	65.5	1.0	690	92.0	0.9
335	36.1	1.3	515	66.5	1.1	695	92.4	0.9
340	37.1	1.1	520	67.0	1.2	700	92.8	1.0
345	38.2	1.4	525	68.1	1.2	705	93.4	1.0
350	39.3	1.2	530	68.6	1.1	710	93.7	1.2
355	40.6	1.2	535	69.6	1.4	715	94.4	1.0
360	41.5	1.0	540	70.3	1.2	720	94.7	1.3
365	42.4	1.1	545	71.4	1.6	725	95.4	1.3
370	43.4	1.1	550	72.0	1.4	730	95.9	1.5
375	44.3	1.1	555	73.3	1.9	735	96.7	1.4
380	45.3	1.0	560	74.0	1.5	740	97.0	1.5
385	46.1	1.1	565	75.4	2.0	745	97.8	1.2
390	47.1	1.2	570	76.0	1.5	750	98.1	1.1
395	48.2	1.3	575	77.3	1.9			

*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 samples.

Table III. Mass Fraction Passing Each Sieve

Run 1			Bottle #								
Sieve	1	9	10	14	18	19	23	27	28	32	
(No.)					Mass Fraction (%)						
25	93.42	93.72	93.49	93.73	93.56	93.46	93.51	93.66	93.53	93.84	
30	80.89	80.89	80.83	80.90	81.11	80.99	80.99	80.96	80.89	80.67	
35	64.02	63.64	63.91	64.07	63.92	63.90	64.02	63.77	63.84	64.06	
40	51.66	51.44	51.59	51.65	51.57	51.57	51.76	51.51	51.59	51.58	
45	41.15	40.78	41.03	40.95	40.91	40.99	41.14	40.80	40.87	41.11	
50	28.77	28.41	28.78	28.83	28.38	28.66	28.64	28.50	28.48	29.21	
60	5.35	4.92	5.56	5.52	5.00	5.37	5.25	5.24	5.09	5.74	
Run 2											Average
25	93.84	93.57	93.40	93.76	93.80	93.48	93.46	93.68	93.63	93.55	93.61
30	80.71	80.73	80.42	80.58	80.85	80.76	80.71	80.85	80.79	80.55	80.80
35	63.94	64.03	63.85	64.01	64.06	63.76	64.08	63.70	63.96	63.88	63.92
40	51.66	51.76	51.59	51.71	51.72	51.48	51.88	51.43	51.69	51.68	51.62
45	40.80	40.92	40.92	40.94	40.91	40.65	41.15	40.46	40.75	40.96	40.91
50	28.42	28.54	28.48	28.40	28.53	28.21	28.49	28.08	28.32	28.50	28.53
60	5.00	5.06	5.35	5.08	5.17	4.99	5.17	5.06	5.08	5.20	5.21

Table IV. Comparison of Nominal and Effective Sieve Openings

Sieve (No.)	Sieve Opening (μm)	
	Nominal	Effective
25	710	708
30	600	594
35	500	496
40	425	410
45	355	356
50	300	303
60	250	239