National Institute of Standards & Technology

Certificate

Standard Reference Material® 1003c

Glass Beads – Particle Size Distribution

This Standard Reference Material (SRM) is intended for use in the evaluation and calibration of equipment used to measure particle size distributions (PSD) in the 20 µm to 50 µm diameter range. Typical instrumentation for PSD determination would be laser light scattering (LLS), sedimentation, electrical sensing zone (ESZ) instruments, and wire-cloth test sieves in the range from No. 400 (38 µm) through No. 635 (20 µm). This size range follows that of the coarser beads of SRM 1004b. The SRM consists of a single bottle containing approximately 28 g of solid spherical soda-lime 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 (mass) distribution was determined using both ESZ and LLS techniques. The certified values and associated uncertainties based on the average of results from the two techniques are given in Table 1 [2]. A comparison of the ESZ and LLS techniques is shown in Figure 1.

Certification Procedure: Two splits from each of ten bottles were analyzed with both the ESZ and LLS techniques to measure the diameter at which a specified percentage of the material is smaller. The percentages are 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, and 95. For each percentage, the only experimental setting that changes between the observations is the bottle of material.

Reference Values: The reference values based on SEM are provided in Table 2 [2]. A NIST reference value is a non-certified value that is the best estimate of the true value; however, the value does not meet NIST criteria for certification and is provided with an associated uncertainty that may reflect only measurement precision and may not include all sources of uncertainty [2]. SRM 1003c was analyzed by scanning electron microscopy (SEM) to provide supplementary data.

Information Values: Information values are provided based on ESZ in Table 3, LLS in Table 4, and on sieve analysis in Table 5 [1]. A comparison of nominal and effective sieve openings is provided in Table 6. A NIST information value is a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value, therefore no uncertainty is provided [1].

Expiration of Certification: The certification of **SRM 1003c** 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 SRM preparation, measurement, and certification were performed by J.F. Kelly of the NIST Ceramics Division.

Statistical analyses were performed A.I. Avilés 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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Electrical Sensing Zone

The ESZ technique for determining powder size distribution uses particles suspended in a conductive fluid such as saline solution. The suspension is drawn through an orifice or aperture separating two electrodes between which an electric potential is applied. A particle passing through the orifice (sensing zone) causes a pulse in the circuit impedance, which is proportional to the particle volume. The measured impedance change is converted to a particle volume using a calibration constant obtained by measuring particles of known volume. For a given aperture of diameter (D), the working range is from 0.02D to 0.60D. For SRM 1003c, an instrument with a 100 µm aperture was used to enable measurements over the range from 2 µm to 60 µm. The conductive fluid was an aqueous solution of 0.8 % mass fraction NaCl. The size distributions measured for the ten bottles of SRM 1003c, selected by stratified random sampling, are given in Table 3.

Laser Light Scattering

LLS also uses a suspension of particles in a fluid. Although the glass beads disperse easily in water, in order to utilize a common sample preparation for the ESZ and LLS measurements, the same saline solution was used for both techniques. Approximately 0.4 g powder was added to 20 ml solution in a cuvet. The cuvet was capped and shaken to disperse the particles. Aliquots of approximately 0.5 ml were taken by pipette to inject into the measuring system. Sufficient aliquots were added until the instrument indicated an acceptable concentration. The LLS technique uses approximations to the Mie scattering theory to convert the measured scattering pattern to particle size distribution. Mie theory includes the influence of diffraction, refraction, reflection and polarization effects. This theory requires that we know the real and imaginary refractive indices of the particles and the suspending medium and that the particles are optically homogeneous, smooth spheres. The use of glass spheres in a transparent medium is a nearly ideal design to satisfy the Mie assumptions. A refractive index of 1.5, with an imaginary component of zero, was used for the soda lime glass. The size distributions measured by LLS, for the same ten bottles measured by ESZ, are given in Table 4. A graphical comparison of the mean distribution measured by LLS with the mean distribution obtained by ESZ is shown in Figure 1.

Certified Values and Uncertainty Determination: The certified values and uncertainty estimates are shown in Table 1. The linear mixed-effects (lme) function from the nonlinear mixed-effects (nlme) library for the language S was used to fit the linear mixed-effects model using the restricted maximum likelihood (REML) estimation method [3,4]. The error analyses for the certified diameter values provided with the SRMs follow recommendations contained in the Guide to the Expression of Uncertainty in Measurement [5] and in NIST Technical Note 1297 [6]. Three sources of error were evaluated in determining the uncertainty values: (1) Method accuracy, (2) Reproducibility of the measurements (i.e. measurement uncertainty in Table 1.), and (3) Bottle to bottle differences in the material (i.e. homogeneity uncertainty in Table 1). The first of these is a Type B uncertainty obtained by analysis of the calibration results using SRM 1961 while the latter two are obtained by the REML analysis. These uncertainty terms are combined using a root sum of squares to obtain a combined standard uncertainty. Since there are sufficient data (>30), a Normal Distribution is assumed and thus the expanded uncertainty values can be obtained by multiplying the combined standard uncertainty by the coverage factor $k = 2$. The value of the standard is believed to lie in the interval defined by the expanded uncertainty with a level of confidence of approximately 95 %.

INSTRUCTIONS FOR USE

SRM bottle must be kept tightly capped with a desiccator pack inside. This is to prevent moisture absorption that can cause clumping of the powder. In applications such as sieving where 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.

For sieve testing of this SRM, the entire bottle-unit of beads should be used. The other techniques require much less material and special care must be exercised when taking subsamples from the SRM bottle. The recommended procedure is to use a microriffler to divide the 28 g sample into subsamples until a suitable subsample mass is obtained.

Sieve Analysis Procedure: The sieve testing was designed to provide reference values for sieve analysis. Eight bottles were selected using a stratified random sampling plan. Mechanical sieving was done following recommendations in ASTM STP 447 [7]. The entire bottle of beads was poured onto the top sieve and the sieves were then shaken for 20 minutes. 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. The mass of beads retained on each sieve was used to calculate, by difference from the total mass, the mass fraction percent that is finer for each sieve, (i.e., the ratio of the mass of beads passing through a sieve to the total mass). The results of sieving for each bottle are given in Table 5 as mass percent of beads passing through each successive screen. Table 6 shows a comparison of the nominal sieve opening with the effective sieve size opening. This is 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 No. 400 screen for all bottles tested was 71.1 %. Interpolation between the 70 % (36.2 µm) and 75 % (37.4 µm) values gives an effective opening of 36.5 µm. This compares with the nominal opening of 38 µm. Each

of the effective diameters is well within the ASTM specification [7] for permissible variation of average opening from the nominal sieve opening.

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) 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 as done 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 to keep the loading below six layers of beads. For use with 76 mm (3 in) diameter 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 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 of beads retained on each sieve was used to calculate, by difference from the total mass, the mass fraction percent that is finer for each sieve. 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 of sieve results and as a method to systematically monitor for changes in screens after service. The compliance of wire cloth sieves according to ASTM E-11 specifications can be tested by contacting the NIST Calibration Program at (301) 975-2200.

Scanning Electron Microscopy Analysis

The cumulative volume (mass) distribution was determined using scanning electron microscopy and based on the measurement of approximately one thousand individual beads from two bottles. The backscatter electron images of the particles were acquired as greyscale image files into a computer via a digital interface. The 2048 × 2048 pixel images were analyzed 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 (geometric mean of major and minor diameters). The pixel to length conversion was calibrated by collecting digital images of the calibrated standard NIST SRM 484e. The SEM results for cumulative mass distribution of the two samples is given in Table 2 as a listing of particle diameter versus cumulative mass fraction with the mass fraction sequenced from 5 % to 95 % in 5 % increments.

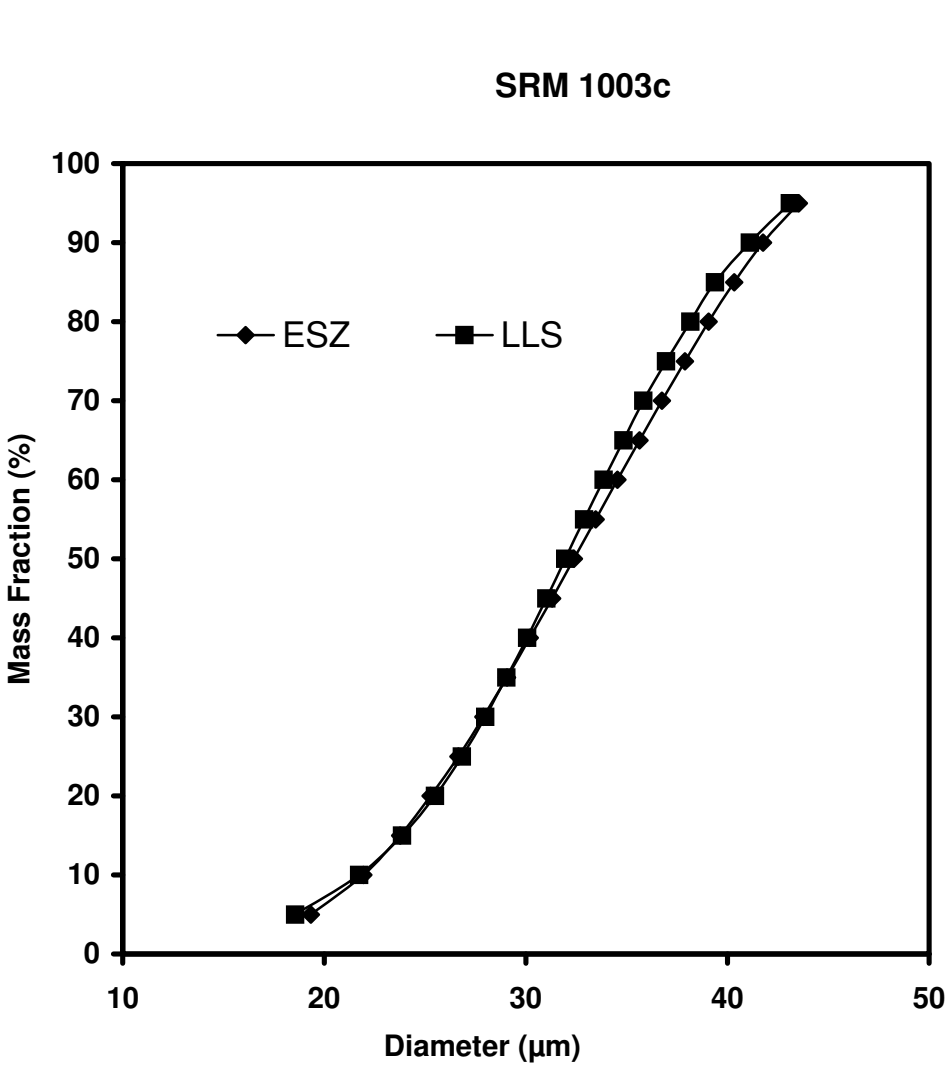

Figure 1. Comparison of the ESZ and LLS results for SRM 1003c.

Table 1. Certified Diameter Values

Cumulative Mass Fraction Finer[a] (%)	Diameter (µm)	Standard Uncertainty Homogeneity (µm)	Standard Uncertainty Measurement (µm)	Type B Standard Uncertainty (µm)	Combined. Standard. Uncertainty (µm)	Expanded Uncertainty[b] (µm)
5	18.9	0.00	0.43	0.41	0.59	1.2
10	21.8	0.15	0.24	0.41	0.50	1.0
15	23.7	0.14	0.26	0.41	0.50	1.0
20	25.3	0.10	0.30	0.41	0.51	1.0
25	26.7	0.10	0.30	0.41	0.51	1.0
30	27.9	0.09	0.27	0.41	0.50	1.0
35	29.0	0.12	0.25	0.41	0.49	1.0
40	30.1	0.12	0.24	0.41	0.49	1.0
45	31.1	0.09	0.26	0.41	0.49	1.0
50	32.1	0.03	0.29	0.41	0.50	1.0
55	33.1	0.00	0.33	0.41	0.52	1.0
60	34.1	0.00	0.37	0.41	0.55	1.1
65	35.2	0.00	0.41	0.41	0.58	1.2
70	36.2	0.00	0.47	0.41	0.62	1.2
75	37.4	0.00	0.48	0.41	0.63	1.3
80	38.6	0.00	0.46	0.41	0.62	1.2
85	39.8	0.00	0.49	0.41	0.64	1.3
90	41.4	0.00	0.36	0.41	0.54	1.1
95	43.3	0.00	0.25	0.41	0.48	1.0

[a] The cumulative mass fraction finer is the portion of SRM 1003c smaller than the certified diameter value.

[b] The uncertainty at each percentile, computed according to the ISO and NIST Guides [4,5], is an expanded uncertainty at the 95 % level of confidence.

Table 2. SEM Data from Two Test Bottles

Cumulative Mass Fraction Finer (%)	Diameter (µm)	Expanded Uncertainty (µm)
5	18.9	0.9
10	22.1	1.1
15	23.8	1.2
20	25.6	1.3
25	27.0	1.3
30	28.2	1.4
35	29.3	1.5
40	30.7	1.5
45	31.6	1.6
50	32.7	1.6
55	33.8	1.7
60	34.7	1.7
65	35.4	1.8
70	36.4	1.8
75	37.6	1.9
80	38.2	1.9
85	39.3	2.0
90	40.5	2.0
95	42.6	2.1

Table 3. Diameters (µm) Measured by Electric Sensing Zone Instrument

Cumulative Mass Fraction Finer* (%)	Bottle Identification 1	2	3	4	5	6	7	8	9	10
	Diameter (µm)									
5	19.3	18.9	19.3	19.3	19.4	19.3	19.7	18.8	19.1	19.0
10	21.9	21.6	21.9	21.9	22.0	21.8	22.2	21.4	21.7	21.5
15	23.8	23.4	23.7	23.8	23.9	23.6	24.1	23.3	23.6	23.3
20	25.2	24.9	25.2	25.4	25.4	25.1	25.6	24.7	25.1	24.8
25	26.6	26.3	26.6	26.8	26.8	26.5	26.9	26.1	26.5	26.2
30	27.8	27.6	27.8	28.0	28.0	27.7	28.1	27.3	27.7	27.5
35	28.9	28.7	29.0	29.2	29.3	28.9	29.3	28.5	28.9	28.5
40	30.0	29.9	30.1	30.4	30.4	30.0	30.4	29.7	30.1	29.7
45	31.1	31.0	31.3	31.4	31.5	31.2	31.5	30.8	31.2	30.8
50	32.2	32.1	32.3	32.5	32.5	32.2	32.5	31.9	32.3	31.9
55	33.3	33.2	33.4	33.6	33.6	33.3	33.6	33.0	33.4	33.0
60	34.4	34.2	34.4	34.7	34.7	34.4	34.6	34.0	34.4	34.1
65	35.5	35.3	35.5	35.8	35.9	35.5	35.7	35.2	35.5	35.2
70	36.6	36.4	36.7	36.9	37.0	36.6	36.9	36.3	36.6	36.3
75	37.8	37.6	37.8	38.0	38.1	37.8	38.0	37.4	37.8	37.5
80	39.0	38.8	39.0	39.2	39.3	39.0	39.2	38.6	39.0	38.7
85	40.2	40.1	40.2	40.6	40.5	40.2	40.4	40.0	40.3	40.1
90	41.7	41.6	41.7	42.0	41.9	41.7	41.8	41.5	41.7	41.6
95	43.5	43.4	43.4	43.6	43.7	43.6	43.6	43.4	43.5	43.4

Table 4. Diameters (µm) Measured by Laser Light Scattering Instrument

Cumulative Mass Fraction Finer* (%)	Bottle Identification 1	2	3	4	5	6	7	8	9	10
	Diameter (µm)									
5	18.8	18.4	18.5	18.6	18.4	18.5	18.6	18.6	19.0	18.3
10	22.1	21.5	21.6	21.8	21.6	21.7	21.8	21.7	22.1	21.4
15	24.2	23.6	23.7	23.9	23.7	23.8	24.0	23.9	24.2	23.6
20	25.7	25.3	25.4	25.5	25.4	25.5	25.6	25.5	25.7	25.3
25	27.1	26.6	26.7	26.9	26.7	26.8	27.0	26.8	27.1	26.6
30	28.2	27.8	27.9	28.0	27.9	28.0	28.1	28.0	28.2	27.8
35	29.2	28.8	28.9	29.1	28.9	29.0	29.2	29.1	29.2	28.9
40	30.3	29.9	29.9	30.1	30.0	30.1	30.2	30.1	30.2	29.9
45	31.2	30.9	30.9	31.1	30.9	31.0	31.2	31.0	31.1	30.9
50	32.1	31.8	31.8	32.0	31.8	32.0	32.1	32.0	32.1	31.8
55	33.0	32.7	32.8	32.9	32.8	32.9	33.0	32.9	33.0	32.8
60	34.0	33.7	33.7	33.9	33.8	33.9	34.0	33.9	33.9	33.8
65	35.0	34.7	34.7	34.9	34.8	34.9	35.0	34.9	34.9	34.8
70	35.9	35.7	35.7	35.9	35.7	35.9	36.0	35.9	35.9	35.8
75	37.1	36.8	36.8	37.0	36.8	37.0	37.1	37.0	37.0	36.9
80	38.2	38.0	38.0	38.2	38.1	38.2	38.3	38.2	38.2	38.1
85	39.4	39.3	39.3	39.4	39.3	39.4	39.5	39.4	39.4	39.3
90	41.2	41.0	41.0	41.1	41.0	41.2	41.2	41.2	41.1	41.1
95	43.1	43.0	43.0	43.1	43.0	43.2	43.1	43.1	43.1	43.1

*The cumulative mass fraction finer is the portion of SRM 1003c smaller than the stated diameter.

Table 5. Sieve Data for Eight Test Bottles
Mass fraction (%) passing through each sieve

U.S.A. Standard Sieve No.	Nominal Opening (µm)	Bottle Identification A	B	C	D	E	F	G	H	Mean (%)
		Mass Fraction Finer (%)								
635	20	7.1	7.5	6.8	6.8	7.2	7.6	8.0	5.8	7.1
500	25	19.5	20.2	17.6	17.8	18.3	16.1	19.5	16.7	18.2
450	32	55.4	52.9	53.0	50.5	48.7	53.2	53.5	51.4	52.3
400	38	74.0	71.3	70.0	69.2	68.7	72.2	74.6	68.8	71.1
325	45	96.7	97.2	96.9	95.9	95.7	96.7	96.8	96.8	96.6
270	53	99.5	99.6	99.6	99.6	99.6	99.6	99.6	99.6	99.6

Table 6. Comparison of Nominal and Effective Sieve Openings

U.S.A. Standard Sieve No.	Sieve Opening Nominal (µm)	Effective (µm)
325	45	44
400	38	36
450	32	33
500	25	25
635	20	20

REFERENCES

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Certificate Revision History: 10 August 2011 (Change of certificate expiration, editorial changes); **16 October 2009** (Extended certificate expiration date and editorial changes); **23 April 2002** (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 http://www.nist.gov/srm.