National Institute of Standards & Technology

Certificate

Standard Reference Material® 1017b

Glass Beads - Particle Size Distribution
(100 µm to 400 µm diameter range)

This Standard Reference Material (SRM) is intended primarily for use in evaluating and calibrating particle size measuring instruments covering the 100 µm to 400 µm range. Typical use is in the evaluation of wire-cloth test sieves in the range of 106 µm (No. 140) through 355 µm (No. 45). A unit of SRM 1017b consists of approximately 70 g of solid spherical soda-lime glass spheres contained in a glass bottle.

Certified Mass Fraction 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 measurand is the cumulative mass distribution of SRM 1017b, determined using both scanning electron microscopy (SEM) and standard sieving procedures on stratified and randomly selected bottles of spheres. The certified values are presented in Table 1, which are the average of results from the SEM analyses of spheres with diameters less than a given length [2]. Metrological traceability is to the SI derived unit for mass fraction (expressed as percent). The uncertainty, computed according to the ISO/JCGM Guide [3], is an expanded uncertainty at the 95 % level of confidence which includes uncertainty due to measurement imprecision as well as material variability.

Information Values: Information values for nominal and effective sieve openings are provided in Table 2, which were used to determine the variability between bottles as well as for a comparison with the SEM results. Figure 1 is a graphical comparison of the SEM and sieve results. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [1]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1017b** 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 Handling and Use"). Periodic recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, mass loss or material spillage that exceeds 2 % of the original mass, or otherwise modified.

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

Coordination of the technical measurements leading to the certification of SRM 1017b was provided by J.F. Kelly formerly of NIST.

Statistical analysis was provided by L.M. Gill formerly of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Certificate Revision History on Last Page

Steven J. Choquette, Acting Director
Office of Reference Materials

INSTRUCTIONS FOR HANDLING AND USE

The entire bottle of spheres should be used in any application of this SRM. If this is impractical, special care must be exercised when producing subsamples from the SRM unit. The recommended procedure is to use a microriffler to divide the entire 70 g unit until an appropriate subsample size is obtained. An appropriate subsample size should be validated by the user; fewer (larger) subsamples are generally preferred.

PREPARATION, AND ANALYSIS

Preparation: The SEM values are based on the measurement of over 9 000 individual spheres from five bottles. Preparation of the SEM samples was designed to achieve a balanced sampling of the different size fractions and a balanced statistical measure of each size fraction. This was achieved by the following steps: five test bottles were sieved into seven size fractions and then riffle split with a spinning microriffler to obtain a sample amount that could be analyzed by SEM. Electron micrographs were taken at seven different magnifications to obtain both adequate counting statistics and diameter resolution for particles in each size range. Images of the glass spheres were captured in a computer interfaced to the SEM. Image analysis software was used for measurement of the sphere diameters. Magnification calibration of the measurement process was determined using SRM 484 *Scanning Electron Microscope Magnification Standard (A Stage Micrometer Scale)* and was checked with a stage micrometer slide calibrated at NIST with the NIST Line Scale Interferometer.

Evaluation of the Effective Opening of Test Sieves with Calibrated Glass Spheres: The allowed variation in sieve openings makes it difficult to compare size determinations made with different sets of sieves even though each set may comply 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 established 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 larger than the average opening can pass through the larger holes. Thus, the effective opening is generally larger than the average opening. 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 openings of sieves are essentially square and particles of irregular shape can pass through openings, even though one of the dimensions of the particle may be 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 spheres is poured onto the top sieve. The sieves are then shaken in exactly the same manner as that to be followed in routine analysis. (To prevent sieve blinding, the spheres should not be used with a single sieve. A rough rule of thumb is to keep the loading below six layers of particles). For use with 76 mm (3 in) test sieves, the mass of spheres must be reduced with a spinning riffler.

After the shaking has been completed, the stack of sieves is disassembled, and the spheres are removed from each sieve and placed into a suitable weighing container. 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 spheres from the sieve and funnel.

Each of the sieve mass fractions is weighed to a precision of at least 0.01 g. After weighing, all spheres are returned to the original bottle and kept for reuse. The mass fraction retained on each sieve is used to calculate the ratio of the mass of spheres passing through a sieve to the total starting mass. The effective size of the sieve opening is then determined by interpolation between the nearest bracketing values given in Table 1.

Note: The above calibration procedure is for use in comparison of sieve results and as a method to periodically monitor for changes in sieves after service. This procedure is <u>not</u> to be used as a certification for test sieves.

Certified Mass Fraction Values: For each diameter, the certified value and expanded uncertainty are provided in Table 1. The uncertainty was calculated according to the ISO/JCGM Guide [3] and is an expanded uncertainty at the 95 % level of confidence, which includes uncertainty due to measurement imprecision as well as material variability.

Table 1. Cumulative Size Distribution
Certified Mass Fraction (w_B) of Spheres with Diameters Less Than the Diameter Indicated for SRM 1017b

Diameter (μm)	w_B (%)	Uncertainty (%)	Diameter (μm)	w_B (%)	Uncertainty (%)
100	2.6	0.3	252	72.8	1.0
104	3.5	0.3	256	73.8	1.2
108	4.8	0.3	260	74.8	0.9
112	6.4	0.3	264	75.8	0.9
116	8.7	0.3	268	76.7	0.9
120	10.8	0.4	272	77.7	0.7
124	13.4	0.3	276	78.6	0.9
128	16.1	0.8	280	79.5	1.0
132	18.7	0.9	284	80.4	1.0
136	21.4	0.9	288	81.3	0.7
140	23.8	0.9	292	82.2	0.5
144	26.1	0.7	296	83.2	0.8
148	28.7	0.6	300	84.2	0.6
152	31.6	0.9	304	85.1	0.8
156	34.3	1.0	308	86.0	0.9
160	36.8	0.9	312	86.6	1.1
164	39.1	0.9	316	87.1	1.1
168	41.3	0.9	320	87.7	1.2
172	43.4	0.9	324	88.3	1.2
176	45.1	0.8	328	88.9	1.4
180	47.0	0.9	332	89.4	1.4
184	48.7	0.8	336	90.1	1.4
188	50.6	0.7	340	90.8	1.3
192	52.3	0.8	344	91.4	1.3
196	54.1	0.7	348	92.0	1.3
200	55.6	0.7	352	92.5	1.4
204	57.2	0.7	356	93.1	1.4
208	58.7	0.8	360	93.8	1.4
212	60.2	0.6	364	94.3	1.3
216	61.6	0.4	368	94.9	1.2
220	62.9	0.6	372	95.5	0.9
224	64.4	0.7	376	96.1	1.2
228	65.7	0.7	380	96.6	1.2
232	66.8	0.7	384	97.1	1.2
236	68.1	0.9	388	97.6	1.2
240	69.4	0.9	392	98.0	1.2
244	70.7	0.9	396	98.4	1.0
248	71.8	0.9	400	98.7	1.0

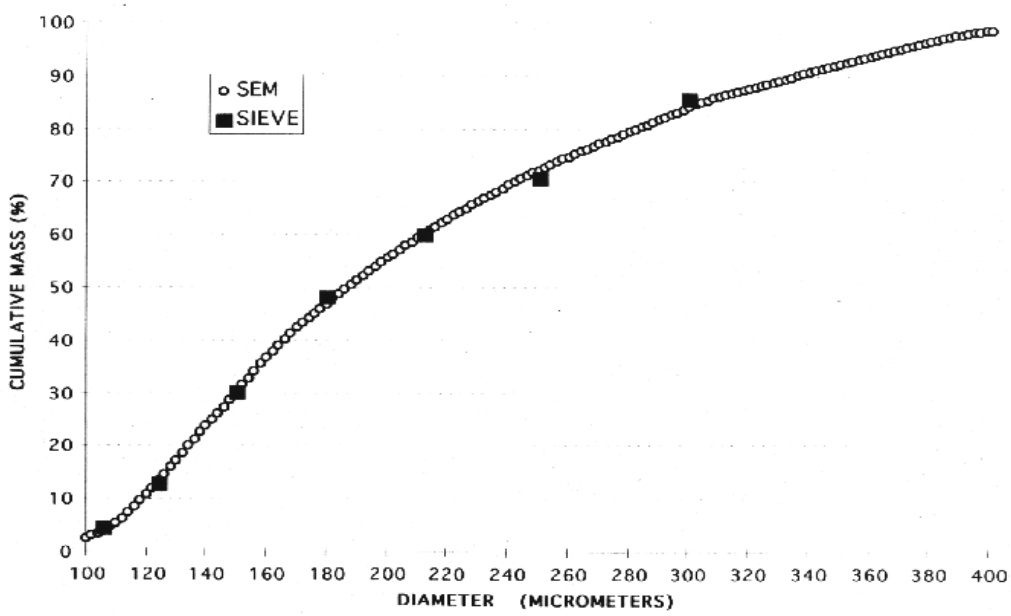

Figure 1. A graphical comparison of the SEM and sieve data results.

Information Values: The values in Table 2 are from a series of sieve analyses following recommended ASTM procedures [4]. A stacked set of seven sieves plus pan were used in a Ro-Tap®(1) sieving unit for a 15 min vibration time. Ten bottles were sieved with an average material loss of 0.03 g from a 72 g bottle. The effective diameters were obtained by comparing the mass fraction of glass spheres passing through a sieve, with the certified diameter for that mass fraction as listed in Table 1. Each of the effective diameters is well within the permissible variation of average opening from the nominal sieve opening.

The mass density of the glass is 2.47 g/cm^3 as determined by helium gas pycnometry for use with Stokes' Law calculations.

Table 2. Comparison of Nominal and Effective Sieve Openings for SRM 1017b

	Sieve Opening (μm)	
U.S. Sieve No.	Nominal	Effective
140	106	107
120	125	124
100	150	150
80	180	183
70	212	211
60	250	244
50	300	306

(1) Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify 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.

REFERENCES

[1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at http://www.nist.gov/srm/publications.cfm (accessed June 2016).

[2] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at http://www.nist.gov/pml/pubs/sp811/preface.cfm (accessed June 2016).

[3] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed June 2016); 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/pml/pubs/index.cfm (accessed June 2016).

[4] ASTM E 11-95, *Standard Specification for Wire Cloth and Sieves for Testing Purposes*; Annu. Book ASTM Stand., Vol. 14.02 (1995).

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Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.