National Institute of Standards & Technology

Certificate

Standard Reference Material 1004a

Calibrated Glass Beads

This Standard Reference Material (SRM) consists of approximately 70 g of solid spherical soda-lime glass beads with diameters covering the 40 to 170 micrometer (μm) range. It is intended for use in evaluating and calibrating particle size measurement instrumentation. Typical use is in the evaluation of wire-cloth test sieves in the range from No. 325 (45 μm) through No. 100 (150 μm). This size distribution is somewhat coarser than the previous version, SRM 1004, and should provide a better overlap of sieve sizes with SRM 1017a.

The cumulative volume (weight) distribution was determined using scanning electron microscopy (SEM) and by the Coulter[1] Principle technique using the Coulter Multisizer IIe. The certified values are the mean of the distributions obtained by the two techniques. The microscopy values were based on the measurement of over 9,000 individual beads from ten bottles. Preparation of the microscopy samples was designed to achieve an equal sampling of several bottles, a balanced sampling of the different size fractions and a balanced statistical measure of each size fraction. This was achieved by the following steps: 1) the ten test bottles were riffle split with a spinning microriffler; 2) the split fractions from each of the bottles were combined to obtain a composite sample; 3) this composite was sieved into seven size fractions; and 4) each of the seven size fractions was riffled to obtain a sample amount which could be analyzed by SEM. Photomicrographs were taken at five different magnifications to obtain both adequate counting statistics and diameter resolution for particles in each size range. Sphere diameters were measured using a digitizing tablet on photoenlarged scanning electron micrographs of the beads. Calibration of the measurement process was done using SRM 1965, Microsphere Slide (10 μm Polystyrene Spheres), and a prototype of the new SRM 2090, Low kV SEM Magnification Calibration Standard.

The certified values are presented in Table I as the weight percentage of beads with diameters less than a given length. At each diameter, the certified value and the uncertainty define a 95% prediction interval. The stated uncertainty includes allowances for measurement imprecision as well as material variability. A 95% prediction interval predicts where the true mean percentile lies for 95% of the samples of this SRM.

Coulter Multisizer IIe measurements were made on two samples from each of the ten bottles and were used to determine bottle to bottle homogeneity as well as for the size distribution certification. The samples for the Coulter analyses were taken from the same riffle splits used for the microscopy samples. Approximately 35,000 beads were measured for each of these twenty analyses. SRM 1961, Nominal 32 μm Diameter Polystyrene Spheres, were used to verify the calibration of the Multisizer IIe. Comparison of SEM and Coulter results is included for informational purposes in Table II.

The overall technical direction and optical measurements leading to the certification were provided by J.F. Kelly of the NIST Ceramics Division. The Coulter measurements were provided at Coulter Corp. by H. Hildebrand. Statistical analysis was performed by L.M. Oakley and S.B. Schiller of the NIST Statistical Engineering Division.

[1]The use of a trademark in this certificate is for identification only and does not imply endorsement of the product by the National Institute of Standards and Technology.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by N.M. Trahey.

Gaithersburg, MD 20899
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Use: In any application of this SRM, the entire bottle of beads should be used for the measurement. However, 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 70-g sample into subsamples and then further divide these subsamples by riffling until a suitable sample size is obtained.

Directions for Using Calibrated Glass Beads for the Evaluation of the Effective Opening of Sieves:
Calibration of test sieves to obtain an effective opening has been described previously.[1] 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. 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 sieve openings are essentially square and particles of irregular shape can pass through, even though one of the dimensions of the particle is considerably larger than the size of the opening. This is especially true for needlelike shapes. The average diameter of such 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 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 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 particle mass. A rough rule of thumb is to keep the loading below 6 layers of particles. Note: 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 pouring 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 container and kept for reuse. The weight percent retained on each sieve is used to calculate the weight percent finer as the ratio of the weight of beads passing through a sieve to the total starting weight. 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 changes in screens after service. This procedure is <u>not</u> to be used as a certification for test screens. The certification of wire cloth sieves is carried out according to ASTM E 11, Specification for Wire-Cloth Sieves for Testing Purposes, or other applicable national test specification.

Table I. Cumulative Size Distribution
Percent of Spheres with Diameters Less Than the Size Indicated

Diameter (µm)	Weight (%)	Uncertainty* (%)	Diameter (µm)	Weight (%)	Uncertainty* (%)
40	2.7	2.2	106	64.9	3.1
42	6.4	2.3	108	68.1	2.9
44	10.9	2.4	110	71.3	2.8
46	15.5	2.2	112	73.6	2.6
48	19.2	2.5	114	75.7	2.4
50	22.2	2.8	116	78.0	2.4
52	24.7	2.9	118	80.2	2.2
54	26.8	3.0	120	82.2	2.1
56	28.9	3.1	122	83.7	2.1
58	31.1	3.3	124	84.6	2.0
60	33.3	3.4	126	85.0	2.0
62	35.3	3.4	128	85.2	2.1
64	36.8	3.5	130	85.3	2.1
66	37.9	3.5	132	85.4	2.1
68	38.4	3.5	134	85.5	2.1
70	38.9	3.5	136	85.6	2.1
72	40.1	3.6	138	85.7	2.1
74	41.8	3.6	140	85.8	2.2
76	43.6	3.6	142	85.9	2.2
78	45.8	3.5	144	86.1	2.2
80	47.8	3.6	146	86.4	2.2
82	49.7	3.6	148	86.9	2.2
84	51.3	3.5	150	87.6	2.4
86	52.9	3.5	152	88.5	2.3
88	54.0	3.5	154	89.6	2.0
90	54.7	3.5	156	91.0	2.0
92	55.1	3.4	158	92.2	1.9
94	55.2	3.4	160	93.4	1.7
96	55.5	3.4	162	94.5	1.5
98	56.0	3.4	164	95.5	1.2
100	57.4	3.4	166	96.6	0.8
102	59.3	3.3	168	97.4	0.7
104	62.0	3.3	170	98.1	0.7

*At each diameter, the certified value and the uncertainty define a 95% prediction interval. The stated uncertainty includes allowances for measurement imprecision as well as material variability. A 95% prediction interval predicts where the true mean percentile lies for 95% of the samples of this SRM.

Table II. Volume % Less Than Diameter Values for
SEM and Coulter Techniques

Volume (%)	Diameter SEM (μm)	Coulter (μm)
10	42.9	44.5
20	47.3	49.6
30	56.3	57.6
40	70.7	72.7
50	82.4	82.4
60	101.8	103.2
70	108.8	109.6
80	117.5	118.1
90	153.5	155.7

The density of the glass is 2.45 g/cm^3 as determined by He gas pycnometry. This value is not certified, but is provided for those wishing to apply Stokes' Law calculations.

REFERENCE

[1]   Carpenter, F.G. and Dietz, V.R., Methods of Sieve Analysis with Particular Reference to Bone Char., J. Res. NBS 45, 328, 1950.