



Certificate

Standard Reference Material[®] 1021

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 2 μm to 12 μm diameter range. Typical methods for PSD determination would be laser light scattering (LLS), electrical sensing zone (ESZ), and sedimentation. The size range of this SRM follows that of the coarser beads of SRM 1003c. A unit of SRM 1021 consists of a single bottle containing approximately 4 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 electric sensing zone and laser light scattering techniques. The certified values and associated uncertainties based on the average of results from the two techniques are given in Table 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, and 90. For each percentage, the only experimental setting that changed between the observations was the bottle of material.

Information Values: Information values based on ESZ and LLS techniques are provided in Table 2 [2]. A comparison of the ESZ and LLS techniques is shown in Figure 1. 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 1021** is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see “Instructions for Use”). Accordingly, periodic recalibration or recertification of this SRM is not required. However, the certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this material over the period of its certification. If substantive technical changes occur that affect the certification, 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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INSTRUCTIONS FOR USE

The sample preparation procedures described here for the certification measurements should be followed to obtain representative sampling and particle dispersion. The LLS and ESZ techniques require much less material than the 4 g provided and special care must be exercised when taking subsamples from the SRM bottle. Representative subsamples of this SRM can be obtained by holding the bottle horizontal and rotating several times before using a spatula to extract about 20 mg powder. Repeat this procedure twice more to extract a total of approximately 60 mg. Add the powder to 20 mL of an appropriate fluid in a cuvet. The cuvet is capped and shaken to disperse the particles, then placed in an ultrasonic bath for 35 minutes to break up any agglomerates. Before sampling, the cuvet is inverted several times to mix the particles in the fluid. Aliquots of approximately 1 mL are taken by pipette to inject into the measuring system. Add sufficient aliquots until the instrument indicates that a sufficient amount has been added to produce an acceptable concentration.

Electrical Sensing Zone Method

The ESZ method 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 1021, an instrument with a $50\ \mu\text{m}$ aperture was used to enable measurements over the range from $1\ \mu\text{m}$ to $30\ \mu\text{m}$. The conductive fluid was an aqueous solution of 0.8 % mass fraction NaCl. The size distributions measured for the ten bottles of SRM 1021, selected by stratified random sampling, are given in Table 2.

Laser Light Scattering Method

The LLS analysis used filtered deionized water to suspend the glass beads. Aliquots of approximately 1 mL were taken by pipette to inject into the measuring system. The LLS technique uses approximations to the Mie scattering theory [3] 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 1.52, 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 2. 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 non - linear 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 [4,5]. The error analyses for the certified diameter values provided with the SRMs follow recommendations contained in the ISO Guide [2]. Three sources of error were evaluated in determining the uncertainty values: 1) method accuracy, 2) reproducibility of the measurements, and 3) bottle to bottle differences in the material. 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 (95 %) uncertainty values can be obtained by multiplying the combined standard uncertainty by $k = 2$.

Table 1. Certified Diameter Values

Cumulative Volume Fraction Finer ^(a) (%)	Certified Diameter (μm)	Standard Uncertainty Homogeneity (μm)	Standard Uncertainty Measurement (μm)	Type B Standard Uncertainty (μm)	Combined Standard Uncertainty (μm)	Expanded ^(b) Uncertainty (μm)
5	2.1	0.01	0.07	0.25	0.26	0.52
10	2.6	0.00	0.09	0.25	0.27	0.53
15	3.0	0.00	0.10	0.25	0.27	0.54
20	3.3	0.00	0.11	0.25	0.27	0.55
25	3.7	0.00	0.12	0.25	0.28	0.55
30	4.1	0.00	0.13	0.25	0.28	0.56
35	4.5	0.00	0.13	0.25	0.28	0.56
40	4.9	0.00	0.12	0.25	0.28	0.56
45	5.3	0.00	0.12	0.25	0.28	0.55
50	5.8	0.00	0.11	0.25	0.27	0.55
55	6.3	0.00	0.11	0.25	0.27	0.54
60	6.8	0.00	0.10	0.25	0.27	0.54
65	7.4	0.00	0.12	0.25	0.28	0.56
70	8.1	0.00	0.15	0.25	0.29	0.58
75	8.9	0.00	0.20	0.25	0.32	0.63
80	9.9	0.00	0.26	0.25	0.36	0.72
85	11.1	0.00	0.35	0.25	0.43	0.86
90	12.9	0.00	0.51	0.25	0.57	1.14

^(a) The cumulative volume fraction finer is the portion of SRM 1021 smaller than the certified diameter value.

^(b) The uncertainty at each percentile, computed according to the ISO Guide [2], is an expanded uncertainty at the 95 % level of confidence.

Table 2. Information Diameter Values (Ten Bottle Averages) Measured by LLS and ESZ

Cumulative Volume Fraction Finer ^(a) (%)	ESZ (μm)	LLS (μm)
5	2.11	2.05
10	2.62	2.50
15	3.04	2.89
20	3.43	3.26
25	3.82	3.63
30	4.21	4.00
35	4.60	4.39
40	5.00	4.80
45	5.41	5.23
50	5.85	5.69
55	6.32	6.20
60	6.83	6.75
65	7.39	7.38
70	8.04	8.09
75	8.81	8.93
80	9.75	9.95
85	10.97	11.26
90	12.77	13.11

^(a) The cumulative volume fraction finer is the portion of SRM 1021 smaller than the stated diameter.

SRM 1021

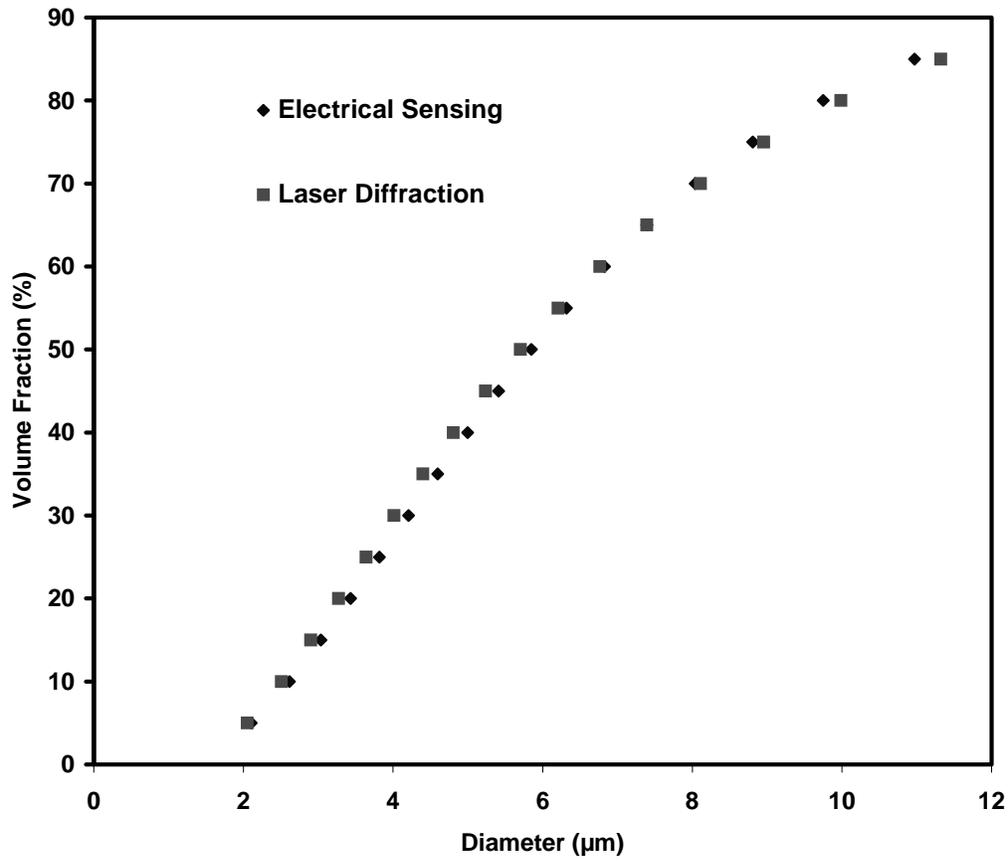


Figure 1. Comparison of the ESZ and LLS results for SRM 1021

REFERENCES

- [1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at <http://www.nist.gov/srm/publications.cfm> (accessed Dec 2011).
- [2] 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 (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Dec 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/pml/pubs/index.cfm> (accessed Dec 2011).
- [3] Searle, S.R.; Casella, G.; McCulloch, C.E.; *Variance Components*; John Wiley & Sons, Inc.: New York (1992).
- [4] Bohren, C.F.; Huffman, D.R.; *Absorption and Scattering of Light by Small Particles*; John Wiley & Sons, Inc.: New York (1983).
- [5] Pinheiro, J.C.; Bates, D.M.; *Mixed-Effects Models in S and S-PLUS*; Springer: New York (2000).

Certificate Revision History: 15 December 2011 (Extension of certification period; editorial changes); 30 September 2009 (This revision reflects an extension of the certification period and editorial changes); 27 February 2008 (Extension of the certification period and editorial changes); 27 March 2003 (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: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.