National Institute of Standards & Technology

Certificate

Standard Reference Material® 1691

Polystyrene Spheres
(Nominal Diameter 0.3 µm)

This Standard Reference Material (SRM) is intended for use as a primary particle size reference standard for the calibration of particle size measuring instruments including electron microscopes. A unit of SRM 1691 consists of one bottle containing approximately 5 mL of a suspension of polystyrene spheres in water at a mass concentration of approximately 0.5 %.

Certified Value: The certified value is given below. The certified values and uncertainties were calculated according to the method described in the ISO/JCGM Guide [1]. The measurand is the number average diameter. Metrological traceability is to the SI unit of length, expressed as micrometers.

Certified Number Average Diameter: 0.269 µm ± 0.004 µm

Information Values: Information values from the original certification of SRM 1691 are provided in Table 1. An information value is considered a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1691** is valid, within the measurement uncertainty specified, until **20 March 2023**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage, and Use"). This certification is nullified if the SRM is damaged, contaminated, or 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.

Overall technical direction and physical measurements leading to the original certification were provided by T. Lettieri, G. Hembree, D. Gilsinn, and E. Marx of the former NIST Mechanical Production Metrology Division. Coordination of the measurements by the cooperating laboratories was performed under the direction of R. Obbink, formerly of NIST.

Overall updated results for this material were provided by J. Dagata, N. Farkas, M. Stocker, P. Kavuri, A. Vladar, and T.V. Vorburger of the NIST Engineering Physics Division and G. Mulholland of the NIST Chemical Sciences Division.

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

David G. Seiler, Chief
Engineering Physics Division

Gaithersburg, MD 20899
Certificate Issue Date: 25 February 2016

Steven J. Choquette, Acting Director
Office of Reference Materials

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INSTRUCTIONS FOR STORAGE AND USE

Storage: Stored SRM 1691 at room temperature. Care should be exercised once the cap has been removed to prevent contamination. Fifty ppm of sodium azide, was added as a biocide before the material was packaged.

Use: Before sampling, manually shake and/or expose the SRM to ultrasonics until the spheres are uniformly distributed. Then take a sample by squeezing a drop from the vial. Use filtered (0.1 µm pore size filter) distilled water for dilution. When electrolytes are used for electrical sensing zone counter measurements, first dilute the sample with water to prevent agglomeration.

ANALYSIS AND CERTIFICATION[(1)]

The number average particle diameter was determined by transmission electron microscopy (TEM) using a reference solution to set the dimensional scale. The value reported is the mean of five independent data sets each consisting of over 100 measurements of 1.0 µm standard spheres and over 30 measurements of nominal 0.3 µm spheres. SRM 1691 was originally calibrated and certified in 1984, before NIST had adopted its current policy on the determination of measurement uncertainty [1]. The original data were reanalyzed [2] in order to produce an uncertainty budget compliant with current NIST policy. Three uncertainty components are considered here:

- u_A – Standard deviation of the means for the set of 0.30 µm particle samples measured
- u_m – Magnification uncertainty, defined as the ratio of the diameters of the 0.3 µm and 1.0 µm particles times the combined standard uncertainty of the 1.0 µm particles
- u_e – Uncertainty in the determination of the point in the particle image that corresponds to the physical edge of the particle

The first component, u_A, is a Type A component estimated by statistical methods. The remaining components (u_m and u_e) are Type B, estimated by other means. These three components are combined in quadrature sum to arrive at the combined standard uncertainty of 0.002 µm for the 0.3 µm particle measurements. Using the Welch-Satterthwaite formula [2], the effective number of degrees of freedom is determined to be 14, giving a coverage factor of 2.14 (equaling a 95 % confidence interval). The final expanded uncertainty for the 0.3 µm (SRM 1691) particle measurements is the combined uncertainty (0.002 µm) multiplied by coverage factor (2.14). The calibrated diameter and its associated expanded uncertainty are shown on page one. The uncertainty is approximately 40 % smaller than the uncertainty given on the original SRM 1691 Certificate.

Information value: The value certified for the number average diameter was confirmed by one additional technique, quasielastic light scattering (QELS). In this technique, the average lifetime of the Brownian motion of the particles suspended in water is measured as a function of scattering angle. This gives a diffusion coefficient which can be used with the Stokes-Einstein relationship to yield the hydrodynamic particle diameter. The uncertainty for the QELS result was also recalculated to make it compliant with the methods described in the ISO/JCGM Guide [1]. The result for the QELS method was: 0.276 ± 0.007 µm. This uncertainty value was obtained by multiplying the QELS combined standard uncertainty (0.0035 µm) by a coverage factor of 1.96, providing a 95% confidence interval.

The size distribution of the polystyrene spheres, as determined by TEM, is narrow with a standard deviation less than 2 % excluding outliers (particles with diameters not on the main peak). The number of small outliers is less than 1 % and the number of large outliers is less than 0.5 %.

[(1)]Certain commercial equipment, instruments, or materials are identified in order 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.

During the original certification, cooperative determinations were performed in the following laboratories:

Brookhaven Instruments Corp., Ronkonkoma, NY, B. Weiner
Eastman Kodak Co., Rochester, NY, D. E. DeCann
Malvern Instruments, Malvern, England, F. McNeil-Watson
G. D. Searle and Co., Skokie, IL, M. Groves

Table 1. Original Certification Information Values for SRM 1691

Method	Laboratory	Number Average Diameter (μm)	Standard Deviation of Distribution (μm)
Electron Microscopy	Kodak	0.248	0.0026
Light Scattering			
Polarization Ratio	Kodak	0.273	0.003
Quasielastic	Kodak	0.272	
Quasielastic	Kodak	0.293	
Quasielastic	Brookhaven	0.273	
Quasielastic	Searle	0.282	0.0032
Quasielastic	Malvern	0.273	
Disc Centrifuge	Kodak	0.25	0.0027
Ultracentrifuge	Kodak	0.28	0.0029

REFERENCES

[1] JCGM 100:2008; *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 Feb 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 Feb 2016).

[2] G. W. Mulholland, *Re-Analysis of the Uncertainty of the 0.895 μm Diameter (NIST SRM 1690) and the 0.269 μm Diameter (NIST SRM 1691) Sphere Standards*, J. Res. Natl. Inst. Stand. Technol. Vol. 110, pp. 27-30 (2005).

Certificate Revision History: 25 February 2016 (Update to uncertainty value; change of expiration date; editorial changes); **01 May 1984** (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) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.