U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 1690

Nominal One-μm Polystyrene Spheres

(In cooperation with the American Society for Testing and Materials)

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 microscopes. The SRM is a suspension of polystyrene spheres in water at a weight concentration of about 0.5%.

The number average particle diameter was determined by measuring the light scattered by the polystyrene spheres suspended in water. The value used for the refractive index of polystyrene was $n(\lambda_{vac} = 632.99) = 1.588$. The diameter was determined from the best fit of Mie light scattering theory to the measured intensity versus angle.

Number Average Diameter, μm	Uncertainty, μm
0.895	± 0.008

The uncertainty includes both random and systematic errors.

The sample-to-sample variability (standard deviation) of the number average diameter, as determined on single drops taken from 20 vials (light scattering measurements of water suspensions and electrical sensing zone counter measurements), was found to be 0.0008 μm.

The value certified for the number average diameter was confirmed by two other measurement techniques. The first of these was by measuring the light scattered by individual spheres (8 were measured) levitated in air. In this technique both the diameter and refractive index are determined by the best fit to light scattering theory. In the second technique the average diameter was determined by optically measuring the row length of particles in two dimensional arrays formed by air drying. Scattering by individual particles: (0.900 ± 0.011 μm). Optical array sizing: (0.900 ± 0.015 μm).

The particle size distribution of the polystyrene spheres (as determined by measurements with a transmission electron microscope) is narrow with a standard deviation of about 0.009 μm, excluding small particles with diameters less than 0.6 μm (about 0.5%) and large single particles with diameters in the range of 2-6 μm (about 0.1%). A discordancy test based on the sample kurtosis was used at the 5% level for rejecting these particles [V. Barnett and T. Lewis, Outliers in Statistical Data, (Wiley, 1978) p. 101]. The particles are spherical with an average deviation from sphericity, $(D_{max} - D_{min})/D_{ave}$, of about 0.006. Measurements with an electrical sensing zone counter and by optical microscopy indicated that about 1.5% of the particles are agglomerated doublets.

The material is expected to have a four year shelf life when stored at room temperature provided the cap on the vial is not removed. Care should be exercised once the cap has been removed to prevent contamination.

Before taking a sample by squeezing a drop from the vial, manually shake and/or expose to ultrasonics until the spheres are uniformly distributed. Use filtered (0.2-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.

The technical direction and physical measurements leading to certification were provided by G. Mulholland, T. Lettieri, G. Hembree, A. Hartman, and E. Marx of the Mechanical Production Metrology Division, with guidance on statistical analysis provided by K. Eberhardt of the Statistical Engineering Division.

The overall coordination of the measurments by the cooperating laboratories was performed under the direction of R. Obbink, Research Associate, ASTM-NBS Research Associate Program.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.K. Kirby.

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(over)

George A. Uriano, Chief
Office of Standard Reference Materials

Cooperative determinations were performed in the following laboratories:

Air Products and Chemicals, Allentown, Pennsylvania, D.J. Nagy
Coulter Electronics Corp., Hialeah, Florida, R.T. Rodewald
Dow Chemical Co., Midland, Michigan, M.A. Langhorst
Duke Scientific Corp., Palo Alto, California, S.D. Duke
Eastman Kodak Co., Rochester, New York, B.C. Wood
General Electric Co., Worthington, Ohio, E.J. Connors
Pacific Scientific, Menlo Park, California, L.D. Carver

The following results are given for information only.

Method	Laboratory	Number Average Diameter (μm)	Standard Deviation of Distribution (μm)
Transmission Electron Microscope	Kodak	0.875	0.018
Opitcal Microscope Array Sizing	Kodak	0.895	- - - - -
Light Scattering			
Polarization Ratio	Kodak	0.900	0.067
Quasielastic	Kodak	0.93[a]	- - - - -
Quasielastic	Coulter	0.896[a]	- - - - -
Light Absorption	Pacific Scientific	0.87	0.030
Electrical Sensing Zone	Kodak	0.89[b]	0.023
Electrical Sensing Zone	Duke	0.90[b]	0.058
Electrical Sensing Zone	Coulter	0.87[b]	- - - - -
Electrical Sensing Zone	G.E.	0.89[b]	0.017
Disc Centrifuge	Kodak	0.91	0.046
Disc Centrifuge	Air Products	0.88	0.26
Hydrodynamic Chromatography	Dow	0.88[c]	- - - - -

[a] Type of average not specified.
[b] Number median diameter.
[c] Volume median diameter.