

National Bureau of Standards

Certificate

Standard Reference Material 1961

Nominal 30- μm Diameter 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 measuring instruments including flow-through counters and optical and electron microscopes. SRM 1961 is a suspension of polystyrene spheres in water at a weight concentration of about 0.5%.

The number average particle diameter was measured in air using Center Distance Finding (CDF), an optical technique related to array sizing [1]. Over 2000 particle diameters were measured with this technique. The certified value is:

Number Average Diameter, μm

29.64

Uncertainty, μm

± 0.06

The uncertainty consists of both random and systematic errors, and includes sample-to-sample variability.

The value certified for the number average diameter was confirmed by one other measurement technique, Metrology Electron Microscopy (MEM). In this technique, which measures the particles in an ultra-high vacuum, the particles are individually scanned through a stationary electron beam while the position of the scanning stage is monitored by a stabilized helium-neon laser interferometer. A plot of secondary-electron intensity vs. stage position gives the particle diameter [2]. The result from MEM is $29.68 \pm 0.05 \mu\text{m}$.

The size distribution of the polystyrene spheres, as determined by CDF, is Gaussian with a coefficient of variation of 0.8% (excluding particles with diameters not on the main peak). The material was precision sieved with a 33- μm diameter-opening electroformed sieve to remove oversized particles. As a result, the number of oversized outliers from the main peak is less than 1%; the same is true for the undersized outliers. A sphere is defined as an outlier if its diameter is more than $4\sigma_D$ from the number average diameter of the main peak. The material is expected to have at least a four-year shelf life when stored at room temperature, provided the cap on the vial is not removed. Care should be exercised to prevent contamination once the cap has been removed. Fifty $\mu\text{g/g}$ of sodium azide was added as a biocide before the material was packaged.

Before sampling, manually shake and/or expose the SRM vial to ultrasonics until the spheres are uniformly distributed, then take a sample by squeezing a drop from the vial. Use filtered (0.4- μm pore size filter) distilled water for dilution. When electrolytes are used for electrical sensing-zone measurements, first dilute the sample with water to prevent agglomeration.

The technology necessary to produce these latex particles was developed by Lehigh University and the National Aeronautics and Space Administration (NASA) during five space shuttle missions in 1982 and 1983. The 30- μm particles in SRM 1961 were manufactured in space aboard the space shuttle Challenger during the NASA STS-11 mission. The particles were provided by NASA for certification by NBS as a Standard Reference Material to be made available to the scientific and technical community.

The technical direction and physical measurements leading to certification were provided by A.W. Hartman, T. Doiron, G.G. Hembree, and T.R. Lettieri of the Precision Engineering Division of the National Bureau of Standards.

Manufacture of the particles was carried out under the direction of J.W. Vanderhoff of Lehigh University and D.M. Kornfeld of the National Aeronautics and Space Administration.

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

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

January 20, 1987
Gaithersburg, MD 20899

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(Over)

[1] A.W. Hartman, **Powder Technology** **46**, 109 (1986).

[2] S. Jensen, G. Hembree, J. Marchiando, and D. Swyt in **Semiconductor Microlithography VI**, SPIE Vol. **275** (SPIE, Bellingham, Wash., 1981).

Cooperative determinations were determined in the following laboratories:

Duke Scientific Corp., Palo Alto, California, S.D. Duke (also performed precision sieving).

Lehigh University, Bethlehem, Pennsylvania, J.W. Vanderhoff.

National Aeronautics and Space Administration, Huntsville, Alabama, D. Kornfeld.

Particle Data Systems, Elmhurst, Illinois, R. Karuhn.

The following results are given for information only:

| <u>Method</u> | <u>Laboratory</u> | <u>Number Average Diameter (μm)</u> | <u>Standard Deviation of Distribution (μm)</u> |
|-----------------------------|-------------------|---|--|
| TEM | Lehigh | 31.31 | 0.39 |
| Electrical- sensing zone | Particle Data | 30.16 | 0.78 |
| | Duke | 29.61 | 0.26 |
| | NASA | 29.09 | 0.37 |
| Optical microscope | Duke | 29.57 | - - - |