National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1964

Nominal 60 nm Diameter Polystyrene Spheres

This Standard Reference Material (SRM) is intended for the calibration/validation of particle sizing instruments, including electron microscopes, differential mobility analyzers, scanning surface inspection systems, and other light scattering instruments. A unit of SRM 1964 consists of 5 mL of polystyrene spheres in deionized filtered (0.2 μm pore size) water. The particle suspension contains primary spheres (monomers) with very few agglomerates. The spheres are present at a mass fraction of approximately 0.5 % and are supplied in a dispensing vial.

The size probability distribution is $P(D) = dN/dD$, where dN is the fraction of particles with diameter between D and $D + dD$. The modal diameter, which is the diameter where $P(D)$ takes the largest value, also called the mode of $P(D)$, was measured for the spheres suspended as an aerosol using differential mobility analysis (DMA) [1]. The certified value for the modal diameter of the polystyrene spheres as an aerosol is given in Table 1.

Table 1. Certified Modal Sphere Diameter and Expanded Uncertainty

Modal Diameter (nm)
60.39 ± 0.63 [(a)]

[(a)] The expanded uncertainty (95 % confidence interval) includes both Type A and Type B uncertainties calculated according to the ISO and NIST Guides [2].

These results are based on three repeat measurements on each of three different dates. There was one calibration measurement made either proceeding or following each of the certification measurements.

The value for the combined uncertainty for the modal diameter is 0.31 nm and the number of degrees of freedom for the uncertainty is 123. This information may be used to estimate the uncertainty when using the spheres for calibrating a particle sizing instrument and estimating the effective degrees of freedom.

Expiration of Certification: The certification of SRM 1964 is valid until **31 December 2013**, within the measurement uncertainty specified, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of Certification: NIST will test for presence of agglomeration in the SRM, and, if changes occur before the expiration of the certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The technical direction and physical measurements were provided by T.A. Germer, Optical Technology Division; G.W. Mulholland, M.K. Donnelly, and S.R. Kukuck, Fire Research Division; V.A. Hackley, Ceramics Division; R.C. Hagwood, Statistical Engineering Division; and D.Y.H. Pui, Particle Technology Laboratory, University of Minnesota.

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The statistical analysis of the data was coordinated by R.C. Hagwood.

The support aspects involved in the preparation and issuance of this SRM were coordinated through the NIST Measurement Services Division.

Traceability: In the present certification measurements [1], the DMA was calibrated using SRM 1963. The certification of SRM 1963 [3] was based upon the certification of the 0.895 µm particle standard, SRM 1690. The certification of SRM 1690 [4], in turn, was based upon the He-Ne laser wavelength in air, 632.807 nm. Lastly, the He-Ne laser wavelength [5] has been determined with respect to the fundamental standard for length. All of the contributions to the uncertainty imposed by this traceability chain have been included in the Type B uncertainty expressed above.

Information Values: An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value.

Additional Information: A single measurement of the size probability distribution $P(D)$ over an extended range of the distribution is given in Table 2 and Figure 1. The standard deviation of the size distribution near the peak is 4.9 nm, and the standard deviation of the entire distribution is 7.9 nm. The number median diameter is 57.5 nm. The number median diameter is the diameter x, at which the integral:

$$\int_0^x P(D)dD = 0.5$$ [1]

The number mean diameter, the volume mean diameter, the light scattering mean diameter, and the dynamic light scattering mean diameter are given in Table 3. The extended measurement of $P(D)$ yielded a slightly different modal diameter than the certified value, because that measurement was only obtained from a single scan on a single day; the difference between those values is statistically insignificant. These are non-certified values with no uncertainty assessed and are provided for information purposes only.

The peak diameter is for the spheres as an aerosol. The contaminants in the suspension result in a residue layer on the spheres. The estimated thickness of this layer is 0.03 nm.

INSTRUCTIONS FOR USE

Storage: Refrigerate the sample (5 °C to 15 °C) but **DO NOT** allow the sample to become frozen. **DO NOT** remove the cap from the vial until the sample is used. It should be noted that no biocide was added during packaging. Once the sample is opened, there is a possibility of biological contamination leading to growth of bacteria or spores. To minimize the occurrence of this unwanted growth, replace the cap immediately after dispensing the sample drops.

Handling and Use: A sample of the spheres may be obtained by squeezing a drop from the vial. Use filtered (0.2 µm pore size filter) deionized water for dilution. Care should be exercised to prevent contamination once the cap has been removed.

The polystyrene spheres may be damaged by exposure to an electron beam. This effect can be minimized by using a short exposure time and low electron flux [6].

[1] That is where the cumulative distribution function is 0.5.

Table 2. Information values on the size probability distribution, $P(D)$, obtained from a single measurement scan over an extended range.

D (nm)	$P(D)$ (nm^{-1})	D (nm)	$P(D)$ (nm^{-1})
10.811	0.00009	59.412	0.06676
15.430	0.00008	60.645	0.06895
19.079	0.00013	61.859	0.06504
22.138	0.00010	63.056	0.05756
24.852	0.00030	64.236	0.04241
27.321	0.00075	65.401	0.02680
29.606	0.00121	66.550	0.01461
31.744	0.00229	67.686	0.00631
33.764	0.00413	68.808	0.00217
35.683	0.00402	69.917	0.00090
37.518	0.00579	71.014	0.00043
39.281	0.00782	72.100	0.00036
40.978	0.00985	73.173	0.00016
42.621	0.01263	74.236	0.00010
44.211	0.01393	75.289	0.00013
45.757	0.01784	77.365	0.00012
47.261	0.01822	79.404	0.00013
48.727	0.02021	80.410	0.00014
50.159	0.02408	81.408	0.00011
51.559	0.02794	83.380	0.00009
52.930	0.03435	85.323	0.00005
54.274	0.03966	87.237	0.00002
55.592	0.04775	89.126	0.00001
56.888	0.05460	90.989	0.00000
58.160	0.06152	92.828	0.00000

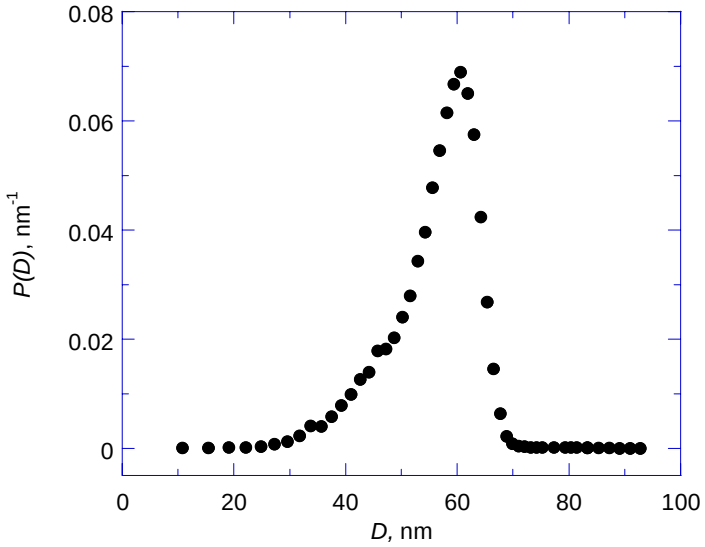

Figure 1. Size probability distribution, $P(D)$, of data provided in Table 2 obtained from a single measurement scan over an extended range.

Table 3. Information value for the weighted mean particle diameter based on the size probability distribution, $P(D)$, given in Table 1.

Peak Diameter [a]	$\langle D_N \rangle$ [b]	$\langle D_V \rangle$	$\langle D_{LS} \rangle$	$\langle D_h \rangle_Z$	$\sigma_{\langle D_N \rangle}$
(nm)	(nm)	(nm)	(nm)	(nm)	(nm)
60.55	55.70	58.49	60.23	59.72	7.90

Number mean diameter: $$\langle D_N \rangle = \int D\, P(D)\, \mathrm{d}D$$

Standard deviation of size distribution: $$\sigma = \left[\int \left(D - \langle D_N \rangle \right)^2 P(D)\, \mathrm{d}D \right]^{1/2}$$

Volume or mass mean diameter: $$\langle D_V \rangle = \frac{\int D D^3\, P(D)\, \mathrm{d}D}{\int D^3\, P(D)\, \mathrm{d}D}$$

Light scattering weighted mean diameter (Rayleigh limit): $$\langle D_{LS} \rangle = \frac{\int D D^6\, P(D)\, \mathrm{d}D}{\int D^6\, P(D)\, \mathrm{d}D}$$

Dynamic light scattering mean diameter [c]: $$\langle D_h \rangle_Z = \left[\frac{\int \frac{1}{D} D^6\, P(D)\, \mathrm{d}D}{\int D^6 P(D)\, \mathrm{d}D} \right]^{-1}$$

[a] The peak diameter is estimated from a cubic fit for the 9 data points within 30 % of the largest value of $P(D) = 0.068419$.
[b] The integrals are evaluated using the trapezoidal rule.
[c] The subscript *h* indicates that this is the hydrodynamic diameter based on the diffusive motion of the particle.

REFERENCES

[1] Mulholland, G.W.; Donnelly, M.K.; Hagwood, C.; Kukuck, S.R.; Hackley, V.A.; Pui, D.Y.H.; *Measurement of 100 nm and 60 nm Particle Standards by Differential Mobility Analysis*; J. Res. Natl. Inst. Stand. Technol., (submitted).

[2] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92–67–10188–9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); 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://physics.nist.gov/Pubs/.

[3] Mulholland, G.W.; Bryner, N.P.; Croarkin, C.; *Measurement of the 100 nm NIST SRM 1963 by Differential Mobility Analysis*; Aerosol Sci. Technol., Vol. 31, pp. 39–55 (1999).

[4] Mulholland, G.W.; Hartman, A.W.; Hembree, G.G.; Marx, E.; Lettieri, T.R.; *Development of a One-Micrometer Diameter Particle Size Standard Reference Material*; J. Res. NBS 90, pp. 3-26 (1985).

[5] Mielenz, K.D. et al; Appl. Phys. Lett., Vol. 7, p. 277 (1965) and Appl. Opt., Vol. 7, p. 289 (1968).

[6] Jung, K.Y.; Park, B.C.; Song, W.Y.; Eom, T.B.; Eom, B.-H.O.; *Measurement of 100-nm Polystyrene Sphere by Transmission Electron Microscope*; Powder Technol., Vol. 126, pp. 255–265 (2002).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.