National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1978

Particle Size Distribution Standard for Gravity Sedimentation

This Standard Reference Material (SRM) is a zirconium oxide powder which is intended for use in the calibration and evaluation of equipment used to measure particle size distribution in the 0.2 to 10 micrometer (μm) range. The SRM consists of a single bottle containing approximately 5 g powder. The characteristics used for selection of this particular zirconium oxide powder were size distribution, shape, and degree of primary particle aggregation. This powder consists of granular, irregular-shaped primary particles with a mean dimension of about 1 μm and a minimal amount of large agglomerates.

The starting material was a 4.5 kg sample from a single lot of Y-TZP zirconium oxide powder produced by St. Gobain/Norton Company and provided by T. Kinisky. This powder was blended to improve homogeneity and split, using spinning rifflers, into 500 bottles averaging 5 g per bottle. A set of eighteen bottles from the 500 was selected at random for sample homogeneity testing. Ten bottles were analyzed at NIST and two bottles each were sent to the four other laboratories for interlaboratory data comparison. Two 0.4 g powder samples were taken with a spatula from each NIST bottle to prepare individual test samples for analysis by SediGraph®. The certified size distribution values given in Table I are based on 40 measurements by the five laboratories. The data from all five laboratories were obtained using x-ray SediGraph® measurements using Model 5000 and 5100 following the procedures specified by NIST.

Certification measurements and technique development were performed by L.H. Lum, NIST Ceramics Division. Statistical analysis was carried out by S.B. Schiller, NIST Statistical Engineering Division.

Participation by A.T. Thornton, Micromeritics Corp. (Norcross, GA 30093); T. Kinisky, St. Gobain/Norton Co. (Northboro, MA 01532); J. Tsubaki, Japan Fine Ceramics Center (Nagoya, Japan); R. Pompe, Swedish Ceramic Institute (S-402 29 Göteborg, Sweden) in the development of this SRM is gratefully acknowledged.

The concept and overall technical direction of this SRM was provided by S.G. Malghan, NIST Ceramics Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by N.M. Trahey.

Gaithersburg, MD 20899
September 16, 1993

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Although the SediGraph® produces a continuous size distribution plot of weight percentage finer than a given diameter, five cumulative percentiles were selected as representative. Analysis from duplicate samples from each of 10 bottles at NIST showed no evidence of material heterogeneity. The certified values in Table I are weighted averages of the laboratory means which included 40 individual percentile measurements.

The measurements were obtained using the Micromeritics Sedigraph® Models 5000 and 5100. Some of the relevant instrument and powder specifications were: starting diameter, 40 µm; powder specific gravity, 5.69 g/cm^3; specific surface area, 5.41 m^2/g; pH of iso-electric point, 8.6. The averages of measurements made at each of the five participating laboratories are included in Table II.

Table I

Certified Size Distribution Data for all Measurements

Cumulative Weight Percentile	Certified Value (µm)	Uncertainty (µm)
10	0.33	0.05
25	0.57	0.05
50	0.98	0.04
75	1.52	0.07
90	2.19	0.17

The uncertainty of each value is defined as a 95% confidence interval and incorporates measurement imprecision as well as lab-to-lab variability.

Table II

Sedigraph® Data for Zirconium Oxide SRM

Laboratory	Stokes Diameter, µm - Cumulative Weight % Finer				
	D_{90}	D_{75}	D_{50}	D_{25}	D_{10}
NIST	2.16	1.51	0.97	0.55	0.30
Micromeritics Instrument Corp.	2.19	1.53	1.00	0.61	0.36
St. Gobain/Norton Company	2.02	1.46	0.98	0.60	0.37
Japan Fine Ceramics Center	2.32	1.55	1.01	0.57	0.31
Swedish Ceramics Institute	2.36	1.61	1.02	0.60	0.35

APPENDIX

Sample Dispersion

Successful use of this SRM depends on following the manufacturer's instructions for Sedigraph® operation and dispersion of the powder as described below. The powder dispersion procedure is similar to the one used for SRM 659, a silicon nitride powder. [1]

A 0.4 g powder sub-sample is removed using a spatula from the vial after shaking and rolling the vial as a precaution against any possible size segregation. The weighed powder is added to 20 cm^3 distilled water in a clean 50 cm^3 glass beaker. The pH of the suspension is adjusted by adding ammonium hydroxide (analytical grade) until the pH is 9.8 ± 0.1. Sonic energy is then applied to the suspension using a probe type ultrasonic disrupter with a 19 mm (0.75 in) diameter probe. The tip of the ultrasonic probe is submerged so that the tip is 10.0 mm above the bottom of the beaker and the output power is adjusted to 40 watts (W). The sonication power is applied for 60 s, and then the power is turned-off for 60 s to prevent overheating of the suspension. The same procedure is repeated for a total ultrasonication time of 3.0 min. The beaker is placed in an ice-water bath during cooling and the suspension is kept stirred to prevent settling and to accelerate cooling to room temperature. The final pH of the suspension is readjusted, if necessary, to 9.8 ± 0.1. Since the pH_{iep} (pH of suspension at which the particles carry a net zero charge) of this powder is at pH 8.6, the dispersion should be prepared at pH 9.8 or above, in order to produce sufficient interparticle repulsion.

REFERENCE

[1] NIST Certificate of Analysis, Standard Reference Material 659, Particle Size Distribution Standard for Sedigraph® Calibration, April 1992.