



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 659

Particle Size Distribution Standard for Sedigraph Calibration

This Standard Reference Material (SRM) is a silicon nitride powder which is intended for use in the calibration and evaluation of equipment used to measure particle size distributions in the 0.2 to 10 micrometer (μm) range. The SRM consists of five (5) glass vials, each containing approximately 2.5 g of powder, sealed in an aluminized mylar pouch. The particle properties used for selection criteria of a particular silicon nitride powder were size, shape, and degree of primary particle aggregation. The selected powder has equiaxed primary particles with a mean dimension of about 1 μm and a minimal amount of large agglomerates.

The starting material was a 5 kg sample from a single lot of SNE-03 silicon nitride powder produced by the Ube Industries, Japan. This powder was blended and split, using spinning riffles, into 2000 glass vials averaging 2.5 g per vial. The final riffle splitting and enclosure in vials were carried out in an argon atmosphere to reduce the risk of surface modification due to atmospheric oxygen and moisture. A set of twenty-two from the 2000 vials was selected for testing sample homogeneity. Twelve of the vials were analyzed at NIST and 2 vials were sent to each of five other laboratories. Two 1-g powder samples were taken from each vial to prepare individual test samples for analysis by Sedigraph [1]. The certified size distribution values given in Table I are based on analysis of the total set of 44 measurements. The data from all six laboratories were obtained by x-ray Sedigraph measurements following the procedures specified by NIST. The averages of measurements made at each of the six participating laboratories are included in Table II.

The concept and overall technical direction of this SRM was provided by S.G. Malghan of the NIST Ceramics Division as a result of involvement in an international interlaboratory comparison program under the auspices of the International Energy Agency. Certification measurements were performed by L.-S.H. Lum, and J.F. Kelly of the NIST Ceramics Division. Statistical analysis was carried out by S.B. Schiller of the NIST Statistical Engineering Division. Participation by B.J. Busovne, Garrett Ceramics Components (Torrence, CA 90509); T. Kinisky, Norton Company (Northborough, MA 01532); A. Prunier, Dow Chemical Company (Midland, MI 48667); T. Tsubaki, Japan Fine Ceramics Center (Nagoya, 456 Japan); and R. Pompe, Swedish Ceramic Institute (S-402 29 Goteborg, Sweden) in the development of this SRM is gratefully acknowledged.

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RESULTS OF STATISTICAL ANALYSIS OF SAMPLE TO SAMPLE AND INSTRUMENTAL VARIABILITY

Although the Sedigraph produces a continuous size distribution plot of weight percentage finer than a given diameter, five cumulative percentiles were selected as representative. The data in Table I are derived by manually reading the percentile values from the chart recorder traces for laboratories using the Sedigraph Model 5000 units and computer derived values for those using the Model 5100. The certified values are an average of the 44 individual percentile measurements.

Table I
Certified Size Distribution Data for all Measurements

<u>Cumulative Weight Percentile</u>	<u>Certified Value (μm)</u>	<u>Uncertainty (μm)</u>
10	0.48	0.10
25	0.81	0.10
50	1.43	0.10
75	2.08	0.11
90	2.80	0.13

The stated uncertainty includes allowances for measurement imprecision and material heterogeneity. Each uncertainty is computed as a 95% prediction interval. The prediction intervals used have the following statistical property: 95% is the statistical expectation of the percentage of all samples of this SRM having cumulative percentiles within the uncertainty limits.

Proper dispersion of the powder is critical in obtaining consistent size distribution measurements. The results of a statistically designed study for evaluation of powder dispersion conditions were used to select the recommended procedure included in the Appendix [2]. The key factors in the preparation of a well-dispersed powder in a suspension are the application of ultrasonic energy to break up agglomerates and the addition of an appropriate surfactant to maintain a stable dispersion.

The measurements were obtained using the Micromeritics Sedigraph Models 5000 and 5100. Some of the relevant instrument and powder specifications were: starting diameter, 50 μm ; and powder density, 3.19 g/cm^3 .

Table II
Sedigraph Data for Silicon Nitride SRM

Laboratory	<u>Stokes Diameter, (μm) - Cumulative Weight % Finer</u>				
	D_{90}	D_{75}	D_{50}	D_{25}	D_{10}
NIST	2.81	2.08	1.42	0.79	0.48
Garrett Ceramic Components	2.80	2.10	1.47	0.86	0.52
Norton Company	2.76	2.10	1.49	0.89	0.54
Swedish Ceramic Institute	2.77	2.02	1.35	0.78	0.48
Dow Chemical Company	2.71	2.05	1.44	0.79	0.43
Japan Fine Ceramics Center	2.85	2.12	1.44	0.81	0.46

REFERENCES

1. Micromeritics Instrument Corp., Norcross, GA 30093.
2. S. G. Malghan, L.-S.H. Lum, J.F. Kelly, E.S. Lagergren and R. Kacker, "Measurement of Particle Size Distribution of Silicon Nitride Powder By Sedigraph", to be published.

Appendix

Sample Dispersion

The sample preparation procedure used in this study is a result of several years of experimentation and round-robin activities under the International Energy Agency, Annex II Subtask 2 and 6. Successful use of this SRM depends on following the manufacturer's instructions for Sedigraph operation and dispersing the powder as described below.

The sample in an entire vial is blended using a small spatula then a 1.0 g powder sub-sample is removed from the vial after shaking and rolling the vial as a precaution against any possible size segregation. A dispersing fluid is prepared by adding 20 mL distilled water and 0.03 g of a 5 wt. % solution of Darvan C (an ammonium polymethacrylate aqueous surfactant obtained from the R.T. Vanderbilt Co., Norwalk, CT 06855) to a clean 50 mL glass beaker. The pH of the water is adjusted by adding ammonium hydroxide (analytical grade) until the pH is 9.0 ± 0.1 . The weighed powder is added to this solution and the pH readjusted to 9.0, if necessary. Sonic energy is then applied to the suspension using a probe type ultrasonic disrupter with a 19 mm (0.75 inch) 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 adjusted to 40 watts (W). (Transmitting 40 W to this load requires an ultrasonic unit a maximum power capacity of about 400 W.) The power is applied for 60 s of sonication, and then the power is turned off for 60 s to prevent overheating. The same procedure is repeated for a total of 3 times. The beaker is placed in an ice-water bath and the suspension is kept stirred to prevent settling and to accelerate cooling.