National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1982

Thermal Spray Powder – Particle Size Distribution
Yttria-Stabilized Zirconia (Spheroidal)

This Standard Reference Material (SRM) is primarily intended for use in the calibration of equipment used to measure particle size distributions (PSD) in the 10 μm to 150 μm range. A unit of SRM 1982 consists of a single bottle containing approximately 10 g of yttria-stabilized zirconia (YSZ) powder.

The PSD values at five mass percentiles were measured by scanning electron microscopy (SEM) and laser light scattering (LLS), and sieving [1]. The certified PSD values by SEM are listed in Table 1. These certified values were determined by the measurement of over 20 000 individual particles from five bottles. The reference PSD values by LLS and sieving are listed in Tables 2 and 3. A comparison of all three methods is shown in Figure 1.

Table 1. Certified PSD Values by SEM

Cumulative Mass (%)	Certified Diameter (μm)	Uncertainty (μm)
10	26.2	± 1.4
25	34.6	± 1.2
50	49.7	± 1.2
75	66.9	± 2.1
90	80.1	± 2.9

Certified Value Uncertainties: The measurand is the diameter for each cumulative mass listed in Table 1. Metrological traceability is to the SI unit for length (expressed as micrometers). The uncertainty at each percentile, computed according to the ISO/JCGM and NIST Guides [2], is an expanded uncertainty at the 95 % level of confidence, which includes uncertainty due to measurement imprecision as well as material variability. Each certified diameter with its expanded uncertainty defines a diameter range within which the associated percentile is expected to lie for at least 95 % of the samples.

Overall technical direction leading to the certification of this SRM were provided by S.G. Malghan and S.J. Dapkunas formerly of the NIST Ceramics Division.

SRM measurement technique, development, and certification were performed by J.F. Kelly and P.T. Pei formerly of the NIST Ceramics Division.

SRM powder characterization was performed by P.T. Pei, J.F. Kelly, and D. Minor formerly of the NIST Ceramics Division.

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Certificate Issue Date: 19 September 2016
Certificate Revision History on Last Page

Steven J. Choquette, Director
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Expiration of Certification: The certification of **SRM 1982** is valid indefinitely, within the measurement uncertainty specified, prior to use provided the unit has not been spilled, contaminated, or otherwise modified. The SRM 1982 powder contains no organic binder and is therefore considered chemically stable. If sieving is performed, it is recognized that some powder will be lost with each use. When the unit's mass loss exceeds 2 % of the original mass, the certification is void. If employing LLS, the unit or subdivision thereof should be discarded after a single use.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Statistical analyses were performed by the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

PREPARATION AND ANALYSIS[(1)]

SRM Preparation and Bottling: The starting material was a 9.08 kg batch of powder from a single lot (95087G-3A) of YSZ produced by Metallurgical Technologies Inc. (Pearland, TX). This powder was chosen for its size distribution, spheroidal particle morphology (Figure 2) and low degree of aggregation. The powder was split using spinning rifflers into bottle units containing approximately 10 g each. A randomized set of 100 bottles from the 730 bottles prepared was selected for homogeneity testing and certification analyses. Ten bottles were sent to Leeds and Northrup Co. (St. Petersburg, FL) for homogeneity determination, (Microtrac® model X-100). The remaining bottles were set aside for use in the SEM microscopy, sieving analysis, and Microtrac round robin study.

Scanning Electron Microscopy Analysis: SEM based image analysis was carried out on six samples using five of the 10 g bottles with two tests run on one bottle. Sample preparation for microscopy entailed both a reduction in the mass of powder and a separation into size fractions. The size fractionation was accomplished by sieving with a Sonic Sifter® (ATM Corp., Milwaukee, WI) using U.S. Standard Series sieves numbers 170 (90 μm), 200 (75 μm), 230 (63 μm), 270 (53 μm), 325 (45 μm) and 400 (38 μm). Subsamples from each of the sieve splits were then produced by successive division using a spinning riffler.

SEM images were acquired for each of the nine sieve fractions. The backscatter electron images of the particles were acquired as greyscale image files into a computer via a digital interface. The 1024 x 1024 pixel images were analyzed to obtain the projected area of each zirconia particle. These areas were converted to a particle volume and particle diameter based on the assumption of spherical particle shape. The pixel-to-length conversion was calibrated by collecting digital images of calibrated standard NIST RM 8090 *SEM Magnification Reference Material* and of a micrometer slide measured at NIST using laser interferometry. The two calibration standards agreed to within a length uncertainty of 1 %. Several hundred particles were measured for each sieve fraction for a total of approximately 4 000 zirconia particles measured from each bottle. Particle size distributions describing the percentage of powder volume represented by particles with diameters less than a given length were calculated using the weighting factors obtained from the sieving results. The diameter values corresponding to the specific mass fractions of 10 %, 25 %, 50 %, 75 %, and 90 % are listed in Table 1. Current practice in the thermal spray industry is to specify these values to define the particle size distribution. A graphical comparison of the mean of the SEM distributions with the mean distributions obtained by sieve analysis and laser light scattering is shown in Figure 1. The diameter values for the sieve analyses were obtained by using the nominal ASTM mesh opening for each sieve.

The sieve results indicated that only about 2 % by mass of the material exceeds 90 μm in diameter in contrast to the result of 20 % obtained by light scattering. This discrepancy is expected to result mainly from the effect of the breaking up of particle clusters by the sonic vibration associated with the sieving.

Laser Light Scattering Analysis by Microtrac Instrument: Nine laboratories participated in this round robin study. Each round robin participant received four bottles for analysis using their model of the Microtrac Instrument. The reference distribution values given in Table 2 are based on 42 measurements by the nine participating laboratories. The data from all nine laboratories were obtained using Microtrac Instrumentation models 7995, 7997, 158704, FRA 9220, and X-100 following the sample preparation procedure specified by NIST. Therefore, in the use of this SRM for LLS, the sample dispersion procedure is mandatory, otherwise the data may not be comparable.

[(1)] Certain commercial equipment, instruments, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Although Microtrac instruments produce a continuous plot of weight percentage finer than a given diameter, five cumulative percentiles were selected as a representative data set, consistent with current industrial practice. Weighted averages of each participant's data are plotted in Figure 3. Analysis from ten samples in duplicate by Leeds and Northrup showed no evidence of material heterogeneity.

Table 2. PSD Reference Values by Laser Light Scattering (Microtrac)

Cumulative Mass (%)	Reference Value (μm)	Uncertainty (μm)
10	24.3	± 0.9
25	36.1	± 1.0
50	53.1	± 1.9
75	73.6	± 3.4
90	95.6	± 5.0

The measurand is the diameter for each cumulative mass listed in Table 2 as determined by LLS. Metrological traceability is to the SI unit for length (expressed as micrometers). The expanded uncertainties, computed according to the ISO/JCGM and NIST Guides [2], include between-laboratories and within-laboratory uncertainty and have a joint level of confidence of 95 %. The reference values and their expanded uncertainties for the five percentiles stated, define a range within which the true percentiles of the distribution are expected to lie with approximately 95 % confidence for all five percentiles considered together.

Sample Dispersion Procedure for Light Scattering Microtrac Method: Use of this SRM for the reference PSD values by the Microtrac method requires rigorous adherence to this sample dispersion procedure. Otherwise, results may not be comparable to the reference values listed in Table 2. Each SRM unit contains 10 g powder sufficient for one PSD analysis by older Microtrac instruments. For newer Microtrac instruments such as model X-100, a smaller sample size is needed and a micro-riffler is recommended for splitting the 10 g sample into subsamples of the desired mass. Another satisfactory splitting method may be used if a micro-riffler is unavailable [3]. Once used, samples should be discarded.

To prepare solutions and measurements, use distilled water for which the pH is adjusted to 9.5 ± 0.1 using 0.1 M sodium hydroxide. Make a paste of sample powder by adding separately prepared 4 % (by mass) sodium pyrophosphate solution at the ratio of 0.5 cm^3 per gram powder. Transfer the paste quantitatively (totally) into the measuring cell containing pH-adjusted distilled water. The transfer of the paste can be achieved by flushing the container with pH-adjusted distilled water.

The size measurement is carried out by following the instrument manufacturer's procedure for instrument operation and determination of PSD.

Sieving Analysis: Ten samples were analyzed by sieving using wire mesh screen numbers 120, 170, 200, 230, 270, 325, and 400. A Sonic Sifter was used in this analysis with the following settings: 10 min at amplitude setting 3, and pulse/shift mode. One bottle of this SRM was used for each test. The measurand is the mass fraction for each sieved mass listed in Table 3 as determined by method indicated. Metrological traceability is to the SI derived unit for mass (expressed as a percent). The stated uncertainties represent one standard deviation from the mean of the mass fractions passing each sieve.

Table 3. PSD Reference Values by Sieving. Mean values for mass passing each sieve.

Mesh (number)	Opening (μm)	Mass Fraction (%)	Standard Deviation (%)
120	(125)	99.93	0.05
170	(90)	97.56	0.56
200	(75)	85.13	1.09
230	(63)	71.06	0.95
270	(53)	55.14	1.07
325	(45)	41.09	1.22
400	(38)	29.87	1.13

The following individuals and companies participated in the development of this SRM:

T. Weigel and P. Plantz; Leeds and Northrup (St. Petersburg, FL).
R.N. Jennings; Alloys International (Baytown, TX).
J. Tan; Hoeganaes Corp. (Riverton, NJ).
J. Cusati; Sulzer Metco (U.S.) Inc. (Hicksville, NY).
M. Roy and C. Kodali; Zircoa Inc. (Solon, OH).
H. Garrelts; Stellite Coatings (Goshen, IN).
R. Douglas; Metallurgical Technologies Inc. (Pearland, TX).
B. Spilka and P. Zajchowski; United Technologies Pratt & Whitney (East Hartford, CT).
L. Burnett; Praxair Surface Technologies (Indianapolis, IN).
C.J. Williams and D. Pohl; Horiba Instruments Inc. (Irvine, CA).
H. Hildebrand; Coulter Scientific Instruments (Hialeah, FL).
J. Bohan; Amherst Process Instruments (Hadley, MA).
C. Dam; Caterpillar Inc. (Peoria, IL).
M. Froning; H.C. Starck Inc. (Newton, MA).

Information Values: Information values are provided in Tables 4 to 6. 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 or only a limited number of analyses were performed. Information values cannot be used to establish metrological traceability.

Table 4. Information Values for Selected Properties of SRM 1982

Specific Gravity by He Pycnometry:	5.86 g/cm^3	±	0.01 g/cm^3
Tap Density:	2.47 g/cm^3	±	0.02 g/cm^3
Hall Apparent Density:	1.82 g/cm^3	±	0.02 g/cm^3
Hall Flow Rate:	No Flow		
Specific Surface Area by Nitrogen Gas Adsorption Method:	0.40 m^2/g	±	0.01 m^2/g

Table 5. Major Chemical Compounds/Components
(data provided by Metallurgical Technologies Inc., Pearland, TX)

Constituent	Mass Fraction (%)
Yttrium Oxide	7.33
Hafnium Oxide	1.39
Silicon Oxide	0.13
Titanium Oxide	0.08
Aluminum Oxide	0.02
Calcium Oxide	0.01
Ferric Oxide	0.02
Magnesium Oxide	0.01
Uranium + Thorium Oxide	0.01
Zirconium Oxide[(a)]	91.00

[(a)]This mass fraction was arrived at by calculation assuming the balance was zirconium oxide.

The averages of measurements made on the zirconia powder at each of the nine participating laboratories are listed in Table 6. The stated uncertainties represent one standard deviation.

Table 6. Industrial Laboratory Data from Round-Robin Study on Zirconia Powder.

	Weighted Average Particle Size (μm)				
Laboratory Microtrac® Model	d_{10}	d_{25}	d_{50}	d_{75}	d_{90}[(a)]
A/X-100	24.5 ± 0.2	36.3 ± 0.4	51.9 ± 0.6	69.1 ± 0.9	89.7 ± 1.6
B/X-100	25.8 ± 0.5	37.3 ± 1.9	55.4 ± 0.5	72.8 ± 0.4	89.6 ± 0.7
C/#7997	23.5 ± 0.2	35.1 ± 0.2	52.6 ± 0.3	72.5 ± 0.4	97.4 ± 0.9
D/X-100	24.5 ± 1.6	36.8 ± 2.4	53.6 ± 2.9	73.8 ± 3.3	94.4 ± 4.0
E/9220-4	24.9 ± 0.1	35.9 ± 0.1	51.1 ± 0.2	69.6 ± 0.3	91.8 ± 0.6
F/7995-12	25.1 ± 0.3	37.6 ± 0.5	55.5 ± 0.4	78.4 ± 1.0	101.9 ± 0.5
G/7995-10	23.7 ± 1.7	36.3 ± 1.8	54.2 ± 2.4	75.9 ± 2.9	96.6 ± 5.6
H/158704-1	23.4 ± 0.1	35.4 ± 0.2	53.5 ± 0.3	75.9 ± 0.7	98.9 ± 2.1
I/SRA#7995-11	24.1 ± 0.4	34.9 ± 0.6	50.6 ± 0.8	74.6 ± 1.7	100.0 ± 1.9
Maximum	25.8	37.6	55.5	78.4	101.9
Minimum	23.4	34.9	50.6	69.1	89.6

[(a)] d_N, is the particle diameter for which N % of the mass is finer.

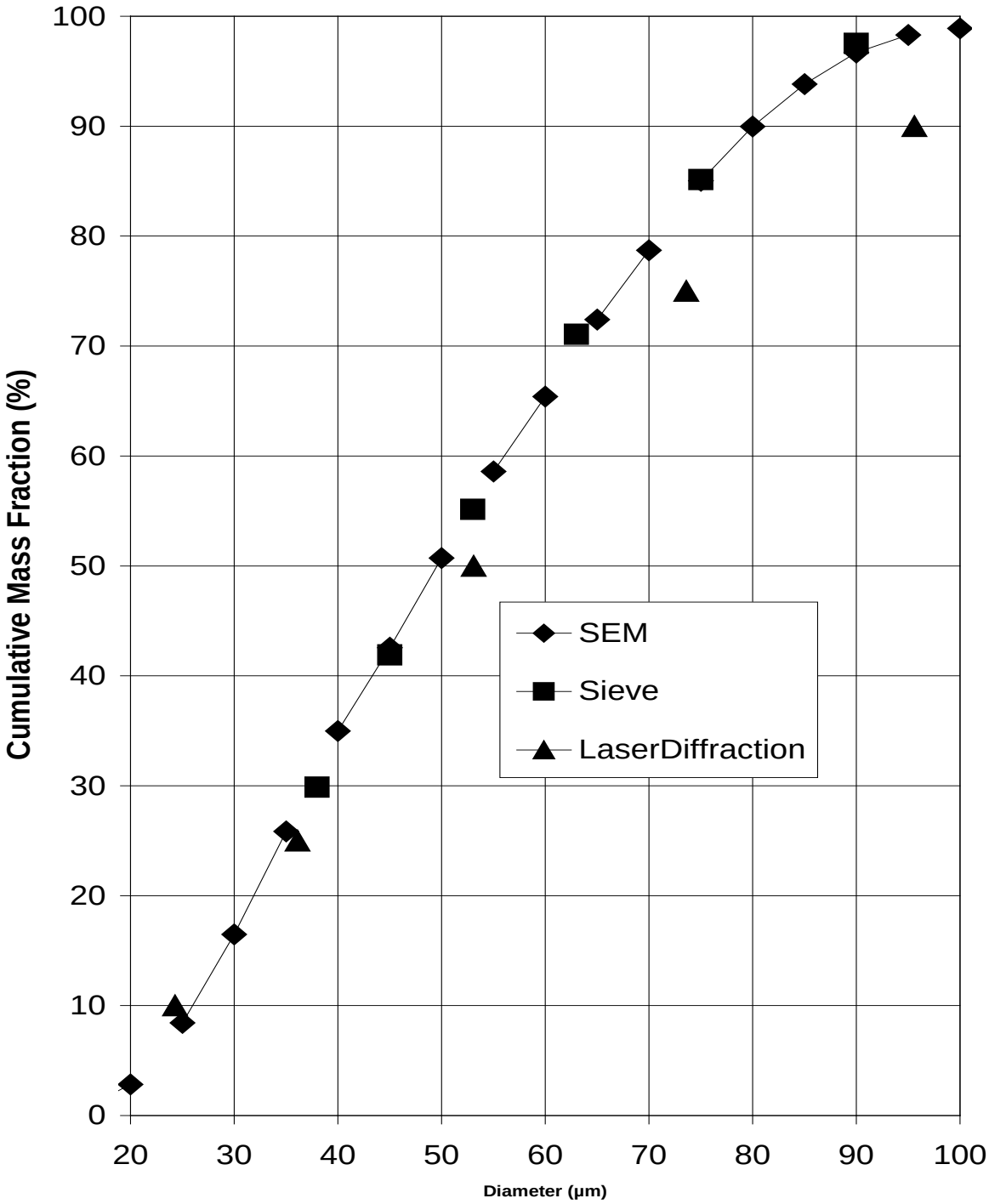

Figure 1. SRM 1982 Size Determination by SEM, Sieving and LLS.

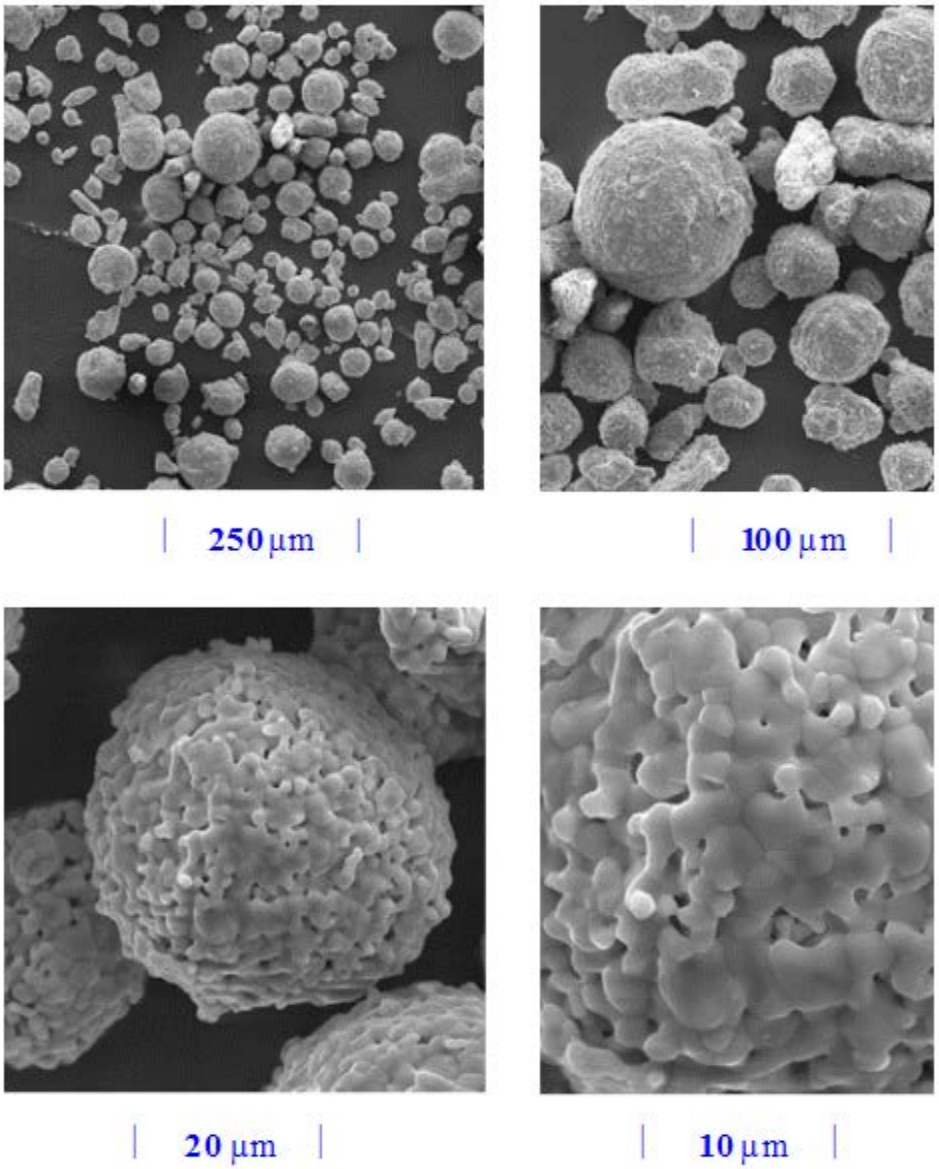

Figure 2. SEM micrographs of SRM 1982 illustrating powder and particle morphology.

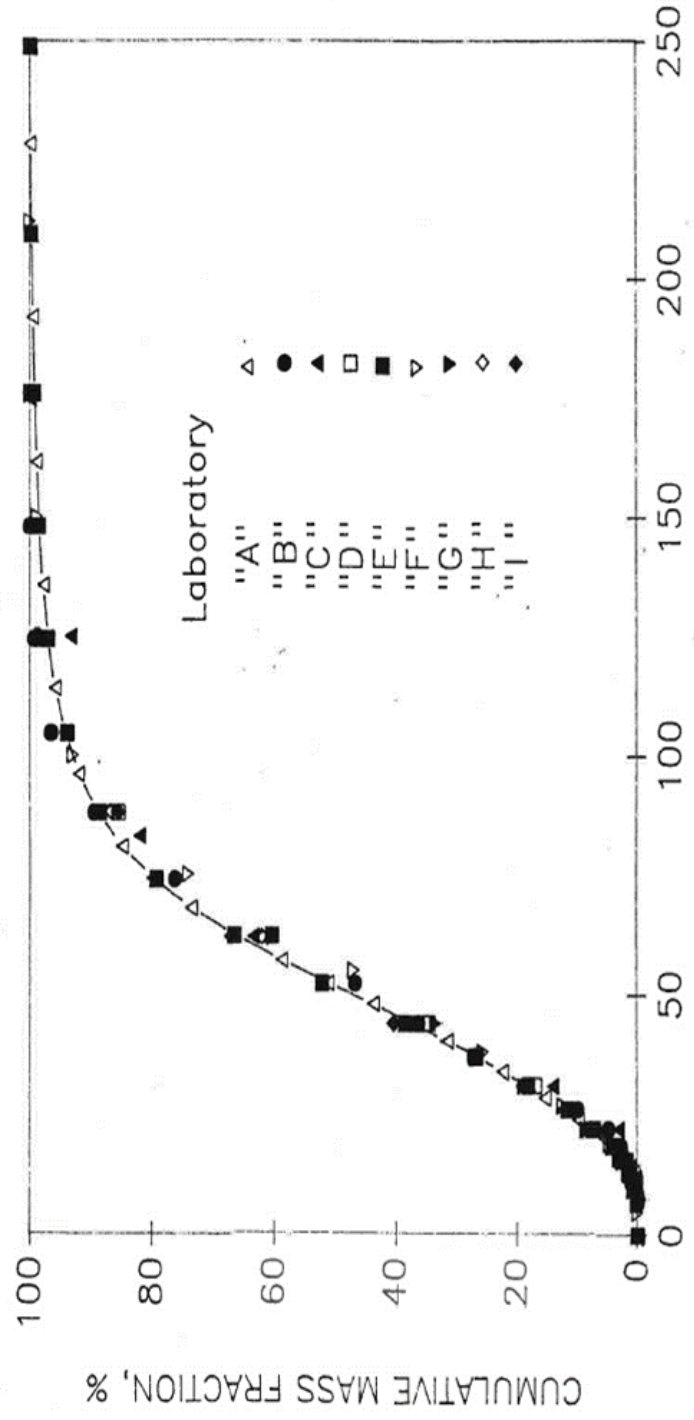

Figure 3. PSD of zirconia by different laboratories using different models of Microtrac®

REFERENCES

[1] Pei, P.T.; Kelly, J.F.; Malghan, S.; Dapkunas, S.; *Analysis of Zirconia Powder for Plasma Spray: Reference Material for Particle Size Distribution Measurement*; Proceeding of the 9th National Thermal Spray Conference, Cincinnati, OH (1996).

[2] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 2016); 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://www.nist.gov/pml/pubs/index.cfm (accessed Sep 2016).

[3] Allen, T.; *Particle Size Measurement*; Powder Technology Series, Ed. B. Scarlett, 3rd Ed., Chapman and Hall: New York, p. 24, (1981).

Certificate Revision History: 19 September 2016 (Editorial changes); 16 September 2003 (This revision reflects the addition of an MSDS); 06 November 1996 (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.