National Institute of Standards & Technology

Report of Investigation

Reference Material® 8988

Titanium Dioxide Powder - Particle Size Distribution

This Reference Material (RM) is intended for use in the evaluation and calibration of equipment used to measure particle size distribution (PSD) values in the 0.1 μm to 0.5 μm particle diameter range. The PSD values were measured using laser light scattering (LLS) and X-ray disc centrifugation (XDC), two common methods for PSD value determination. A unit of RM 8988 consists of a single bottle containing approximately 6 g of rutile titanium dioxide powder.

Reference Values: The cumulative mass distribution values were determined using both LLS and XDC techniques. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification [1]. The reference diameter values and associated uncertainties are specified separately for the two techniques and are given in Tables 1 and 2. A comparison of the results from the two techniques is shown in Figure 1.

The uncertainty was calculated using a linear mixed-effects (lme) function from the non-linear mixed-effects (nlme) library for the computer language S to fit the lme model using the restricted maximum likelihood (REML) estimation method [2,3]. The error analyses for the reference diameter values provided with the RM follow recommendations contained in the ISO/JCGM Guide [4]. Three sources of error were evaluated in determining the uncertainty values: (1) method accuracy; (2) reproducibility of the measurements; and (3) bottle–to-bottle differences in the material. The first of these errors is a Type B uncertainty estimated to be ± 5 % in diameter, while the latter two errors are obtained by the REML analysis. These uncertainty terms are combined using a root sum of squares to obtain a combined standard uncertainty. As there were sufficient data (>30), a normal distribution was assumed, and the expanded (95 %) uncertainty values were obtained by multiplying the combined standard uncertainty by $k = 2$.

Information Value: The mass density of the particles is 4.2 g/cm^3 based on helium pycnometer measurements.

Expiration of Value Assignment: RM 8988 is valid indefinitely, within the measurement uncertainty specified, provided the RM is handled and stored in accordance with instructions given in this Report of Investigation (see "Instructions for Storage"). Accordingly, periodic recalibration or verification of this RM is not required. This report is nullified if the RM is damaged, contaminated, or otherwise modified.

Maintenance of RM: NIST will monitor this RM over the period of its validity. If substantive technical changes occur that affect the value assignment, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

RM preparation and measurements were performed by J.F. Kelly and A. Jilla of the NIST Materials Measurement Science Division.

Statistical analyses were performed by A.I. Avilés of the NIST Statistical Engineering Division.

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

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Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
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INSTRUCTIONS FOR STORAGE

Storage: The RM bottle should be kept tightly capped under dry conditions to prevent moisture absorption that could cause clumping of the powder.

SAMPLE PREPARATION AND MEASUREMENT PROCEDURE[(1)]

Two samples from each of ten bottles were analyzed using both the LLS and XDC techniques to measure the diameter at which a specified percentile of the material is smaller, called the cumulative mass fraction finer. The percentiles are 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, and 90. For each percentile, the only experimental setting that changed between the measurements by each technique was the bottle of material. For each cumulative mass fraction finer, the measured particle diameter values for the ten bottles were averaged to give a single reference diameter value.

LLS Technique: Approximately 0.4 g of the RM powder was added to 40 mL of a solution containing 0.01 % (mass fraction) tetrasodium hexametaphosphate in filtered, deionized water. The powder was dispersed using an ultrasonic probe instrument operating at a power of 40 W in two 2-minute cycles; the resulting suspension was cooled to room temperature between cycles using a cooled water bath.

The particle size measurements were made using a Malvern Mastersizer 2000 laser-based light scattering system. Aliquots containing approximately 1 mL of the powder suspension were extracted using a pipette and added until the instrument indicated that an acceptable concentration had been obtained for analysis. The LLS technique uses approximations to the Mie scattering theory [5] to convert the measured scattering pattern to particle size distribution. Mie theory includes the influence of diffraction, refraction, reflection, and polarization effects. This theory also uses the real and imaginary refractive indices of the particles and the suspending medium and assumes that the particles are optically homogeneous smooth spheres. A refractive index with a real component of 2.5 and an imaginary component of 1.5 was used for the titanium dioxide.

The mean particle diameter for each cumulative mass fraction finer for the LLS measurements is given in Table 1 and plotted in Figure 1.

XDC Technique*:* Approximately 1.0 g of the RM powder was added to 50 mL of a solution containing 0.01 % or 0.05 % (mass fraction) sodium hexametaphosphate in filtered, deionized water. The powder was dispersed in suspension using an ultrasonic probe instrument and cooled as described above.

The particle size measurements were made with 25 mL of the powder suspension, prepared as described above, using a Brookhaven Instruments Corp. Model BI-XDCW X-ray disc centrifuge system. The X-ray source and detector were radially scanned across the disc while the suspension was spun at 157.1 rad/s (1500 rpm) for 20 minutes. For each measurement time, the particle diameter was calculated using the Stokes equation for the settling of particles in laminar flow under the influence of a centrifugal field [6]. The density of the suspension medium was obtained from tables of the variation of water density with temperature and the density value (see "Information Value" above). Note: Users of RM 8988 should use this density value when calibrating the operation of an XDC instrument.

The mean particle diameter for each cumulative mass fraction finer for the XDC measurements is given in Table 2 and plotted in Figure 1.

[(1)] Certain commercial equipment, instruments, or materials are identified in this report 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 are necessarily the best available for this purpose.

Table 1. Reference Diameter Values and Expanded Measurement Uncertainties as Measured by LLS

Cumulative Mass Fraction Finer (%)	Reference Diameter (µm)		
5	0.15	±	0.02
10	0.17	±	0.02
15	0.19	±	0.02
20	0.21	±	0.02
25	0.22	±	0.02
30	0.24	±	0.03
35	0.25	±	0.03
40	0.27	±	0.03
45	0.28	±	0.03
50	0.30	±	0.03
55	0.32	±	0.03
60	0.34	±	0.04
65	0.35	±	0.04
70	0.37	±	0.04
75	0.40	±	0.04
80	0.42	±	0.04
85	0.45	±	0.05
90	0.49	±	0.05

Table 2. Reference Diameter Values and Expanded Measurement Uncertainties as Measured by XDC

Cumulative Mass Fraction Finer (%)	Reference Diameter (µm)		
5	0.16	±	0.02
10	0.18	±	0.02
15	0.19	±	0.02
20	0.21	±	0.02
25	0.22	±	0.02
30	0.23	±	0.02
35	0.24	±	0.02
40	0.25	±	0.03
45	0.26	±	0.03
50	0.27	±	0.03
55	0.28	±	0.03
60	0.30	±	0.03
65	0.31	±	0.03
70	0.32	±	0.03
75	0.34	±	0.04
80	0.36	±	0.04
85	0.39	±	0.05
90	0.44	±	0.05

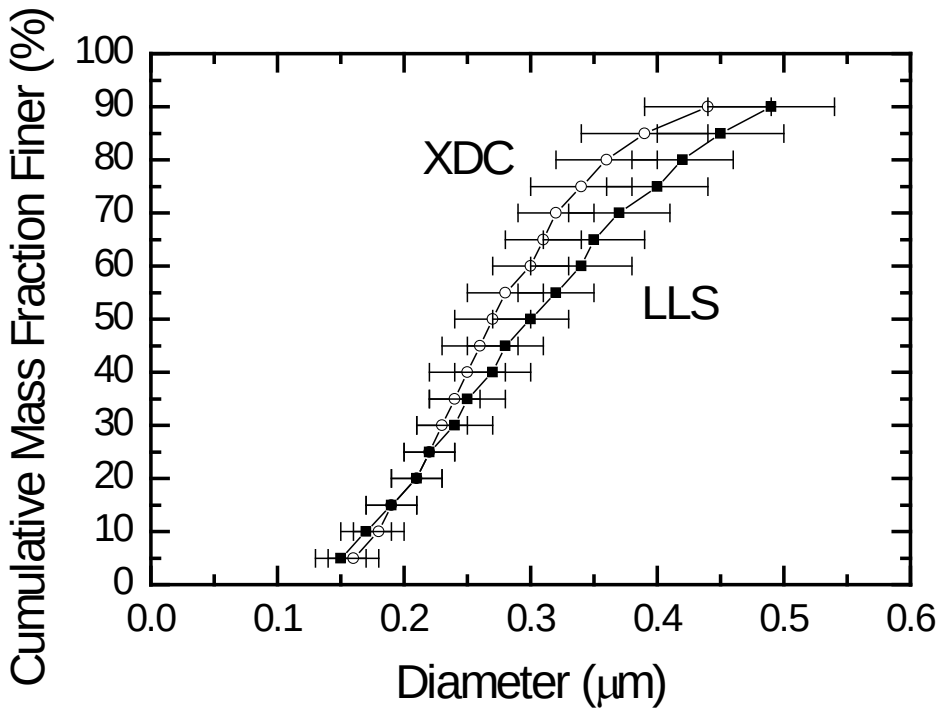

Figure 1. Comparison of the reference particle diameter values as measured by LLS (full symbols) and XDC (open symbols). The symbols represent the means and expanded uncertainty diameter values at each cumulative mass fraction.

REFERENCES

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Certificate Revision History: 24 June 2013 (Removed value assignment expiration date; revised tables 1 and 2 to only include values and expanded uncertainty; editorial changes); 25 March 2010 (Original certificate date).

Users of this RM should ensure that the Report of Investigation 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.