



# National Institute of Standards & Technology

## Report of Investigation

### Reference Material 8632

#### Ultra Fine Test Dust (UFTD)

This Reference Material (RM) is intended to be used as a secondary calibration material for calibrating particle sizing instruments, especially optical particle counters. RM 8632 consists of 20 g of a naturally occurring irregularly shaped mineral dust. The dust is heterogeneous in composition and polydisperse with respect to size.

RM 8632 may be used in conjunction with either of the following two documents: the National Fluid Power Association (NFPA) method T2.9.6 R2 "Hydraulic Fluid Power - Calibration Method of Liquid Automatic Particle Counters" [1] or the International Standards Organization (ISO) method ISO/DIS 11171 "Hydraulic Fluid Power - Calibration of Liquid Automatic Particle Counters" [2].

**Expiration of Material:** The material comprising this RM should remain stable indefinitely. The reference values remain valid provided the RM is handled and used in accordance with the instructions and caution given in this report. However, the size distribution may be altered and the RM invalidated if the material is contaminated or sampled improperly.

**Caution to User:** Scoop sampling directly from the bottle is prohibited because it can result in a non-representative (size fractionated) sample that will permanently alter the size distribution of the remainder of the RM bottle.

**Instructions for Use:** The entire bottle of dust should be used in any application of this RM. If this is impractical, special care must be exercised when taking subsamples from the RM bottle. To subsample, follow an accepted procedure including spin riffing, flat pancake sampling, or cone and quartering [3-5]. These sampling procedures require the entire bottle to be utilized in the reduction to arrive at a split aliquot for analysis.

**Material Source and Processing:** This material was manufactured and donated by Powder Technologies, Inc., Burnsville, MN. RM 8632 is a derivative of Arizona Road Dust (ISO Ultra Fine Dust [6]<sup>1</sup>) and was taken from the same production lot, number 4476J, used in the NFPA round robin summarized in Table 2. Approximately 4.4 kg of material was spin-riffled, bottled, and sealed in containers holding approximately 20 g aliquots by Laboratory Quality Services International, South Holland, IL.

The overall technical direction of this project was provided by R.A. Fletcher of the NIST Surface and Microanalysis Science Division.

Sample preparation and optical particle counting were provided by E.S. Windsor; data analysis was performed by R.A. Fletcher, both of the NIST Surface and Microanalysis Science Division.

Experimental design and statistical review of the data were provided by W.S. Liggett of the NIST Statistical Engineering Division.

The technical and support aspects involved in the issuance of this RM were coordinated through the Standard Reference Materials Program by R.J. Gettings.

Gaithersburg, MD 20899  
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19 Jun 98 (original certificate date)  
\*Editorial change.

Thomas E. Gills, Chief  
Standard Reference Materials Program

<sup>1</sup> Certain commercial equipment, instruments, or materials are identified in this report to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

**Homogeneity Testing and Reference Value Determination:** NIST evaluated the bottle-to-bottle homogeneity and determined the reference values by examining eight bottles of RM 8632 selected from the four quadrants of the spin-riffle sampling wheel. Each 20 g bottle was further spin riffled at NIST to produce approximately 300 mg samples of dust. The material was suspended in clean MIL-H-5606 hydraulic oil to produce suspensions of known concentration at a nominal 3.7 mg/L level. Relative measurements were made using a HIAC/ROYCO optical particle counter (HR-LD 150) equipped with a light extinction sensor calibrated to ISO 4402:1991. No evidence for bottle-to-bottle heterogeneity was observed within the measurement uncertainty calculated.

An optical particle counter calibrated to the existing ISO 4402:1991 (AC Fine Test Dust) method was used to determine the particle size distribution for the eight bottles described above. Table 1 contains the particle size defined by ISO 4402:1991, the mean value of the number of particles counted greater than the specified size ( $n = 29$ ), and the standard deviation of these 29 measurements [7]. The values presented in Table 1 are based on hydraulic oil suspensions with UFTD particle concentrations of 1 mg/L. Optical particle counter measurements on this RM will depend partially upon the calibration of the counter and the particle concentration of the suspension. ISO 4402:1991 is under revision, the replacement method is ISO/DIS 11171 using NIST SRM 2806, because AC Fine Test Dust is no longer commercially available and there is a recognized inaccuracy in ISO 4402:1991 for the small particle end of the distribution [8].

#### REFERENCES

- [1] NFPA T2.9.6 R2-19xx. "Hydraulic Fluid Power - Calibration of Liquid Automatic Particle Counters." National Fluid Power Association, Milwaukee, WI. Revision of ANSI/(NFPA)T2.9.11-1989.
- [2] ISO/DIS 11171, "Hydraulic Fluid Power - Calibration of Liquid Automatic Particle Counters," International Organization for Standardization, Geneva, Switzerland, Proposed revision of ISO 4402:1991, "Hydraulic Fluid Power-Method Using Classified AC Fine Test Dust Contaminant."
- [3] Allen, T. *Particle Size Measurement*. Chapman and Hall, London, p. 23, (1974).
- [4] Pitard, F.F. *Pierre Gy's Sampling Theory and Sampling Practice* CRC Press, Ann Arbor, pp. 240-241, (1995).
- [5] ASTM C 702-93. "Standard Practice for Reducing Samples of Aggregate to Testing Size," American Society for Testing and Materials, West Conshohocken, PA.
- [6] ISO 12103 "Road Vehicle - Test Dust For Filter Evaluation Part I - Arizona Test Dust," International Organization for Standardization, Geneva, Switzerland.
- [7] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and 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).
- [8] Masbaum, G. "Über die Kornverteilung von ACFTD oder Stimmt die ISO 4402?" *Ölhydraulik und Pneumatik*, 25:10, p. 777 (1981).

*Users of this RM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov), or via the Internet <http://ts.nist.gov/srm>.*

Table 1. Reference Mean Values of the Cumulative Number of Particles per mL Greater than Indicated Size for a 1 mg/L Suspension Measured by an Optical Particle Counter Calibrated to ISO 4402:1991.

Size <sup>a</sup> ( $\mu\text{m}$ )	Mean Concentration (particles/mL)	Standard Deviation (particles/mL)
1	4892	136
2	4113	104
3	2990	65.6
4	2085	41.8
5	1397	24.9
6	907.7	14.1
7	570.0	8.64
8	352.4	5.57
9	218.9	3.75
10	134.5	2.53
11	81.98	1.64
12	47.37	1.34
13	27.34	0.72
14	16.62	0.57
15	10.29	0.39
16	6.63	0.25
17	4.43	0.22
18	3.09	0.19
19	2.23	0.12
20	1.66	0.11

<sup>a</sup> Particle size obtained using the ISO 4402:1991 calibration.

Table 2. Participating laboratories, particle counters and optical sensors used for NFPA Round Robin. Sensors calibrated according to ISO 4402:1991. Values for particle concentration (particles/mL greater than indicated size) are presented for information only.

Laboratory	Particle Counter	Sensor	Particle Concentration (particles/mL)					
			> 1 $\mu\text{m}$ <sup>a</sup>	> 2 $\mu\text{m}$	> 5 $\mu\text{m}$	> 7 $\mu\text{m}$	> 10 $\mu\text{m}$	> 15 $\mu\text{m}$
			> 4.2 $\mu\text{m}$ <sup>b</sup>	> 4.6 $\mu\text{m}$	> 6.4 $\mu\text{m}$	> 7.7 $\mu\text{m}$	> 9.8 $\mu\text{m}$	> 13.6 $\mu\text{m}$
Nelson Industries, Inc.	Climet C11000	RLV2 100H	5214	4081	1346	538	119	-----
Nelson Industries, Inc.	HIAC 9064	HRLD 150	5981	4823	1460	554	127	13
Fluid Technologies, Inc.	HIAC 8000	HRLD 400	-----	4872	1564	619	124	7.8
Pall Corp.	Met One 214	Met One 211W (1 to 120 micrometer)	5022	4209	1431	-----	147	16
Pall Corp.	Met One 214	Met One 211W (0.5 to 25 micrometer)	5030	4215	1463	-----	121	14

<sup>a</sup> Size determined following ISO 4402:1991.

<sup>b</sup> Adjusted values resulting from NIST calibration to SRM 2806.

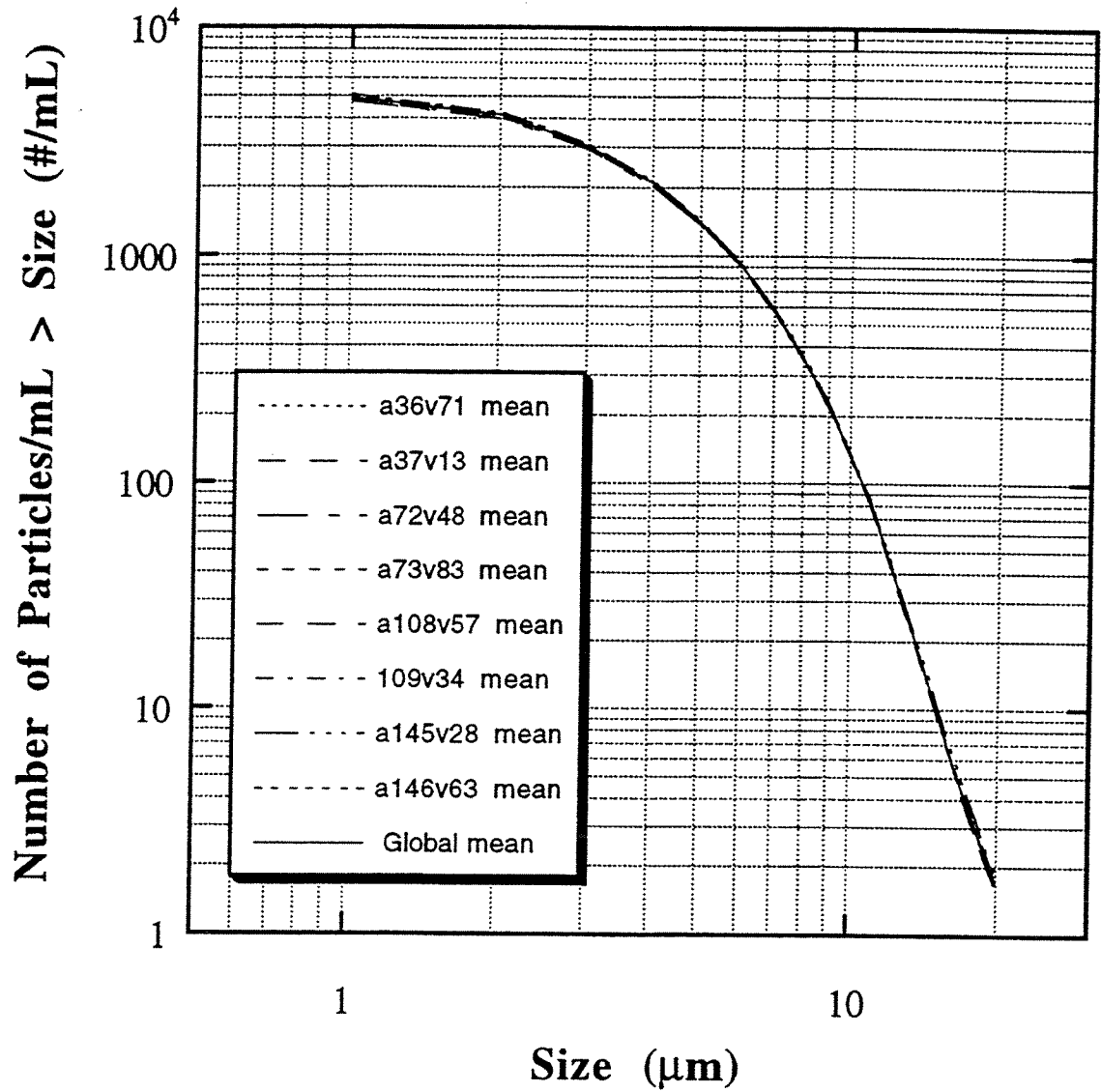


Figure 1. Cumulative number of particles per mL greater than the specified projected area diameter for eight bottles of RM 8632 Ultra Fine Test Dust (UFTD) measured by an optical particle counter calibrated to ISO 4402 :1991.