



National Institute of Standards & Technology

Report of Investigation

Reference Material 8631

Medium Test Dust (MTD)

This Reference Material (RM) is intended to be used as a secondary calibration material for calibrating instrument response of particle sizing instruments, especially optical particle counters. RM 8631 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 8631 can 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 of Liquid Automatic Particle Counters" [1] or the International Organization for Standardization (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: Ideally 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 8631 is a derivative of Arizona Road Dust (ISO Medium Dust [6] also known as PTI 5-80 Test Dust¹ and SAE 5-80 Test Dust) and was taken from the same production lot, No. 4390C, used to make SRM 2806 Medium Test Dust (MTD) in Hydraulic Fluid. Approximately 4.4 kg of material was spin-riffled, bottled, and sealed in containers holding 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.

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

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

Experimental design and statistical review of the data were provided by W.S. Liggett of the NIST Information Access and User Interface Division

Homogeneity Testing and Reference Value Determination: NIST evaluated the bottle-to-bottle homogeneity and determined the reference values by examining eight bottles of RM 8631 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 3.9 mg/L level. Relative measurements were made using an optical particle counter (HIAC/ROYCO HR-LD 150) equipped with a light extinction sensor calibrated to ISO 4402:1991. No evidence for bottle-to-bottle inhomogeneity 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 MTD 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, 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, "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).

Table 1. Reference Mean Values of the 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 ^a	Mean	Standard
(μm)	Concentration (particles/mL)	Deviation (particles/mL)
1	1802	30.0
2	1527	23.7
3	1176	16.2
4	907.7	12.2
5	697.5	10.8
6	532.6	9.25
7	401.4	8.17
8	302.6	6.88
9	233.3	6.10
10	181.8	5.37
11	143.7	4.57
12	114.8	4.38
13	91.04	3.77
14	74.61	3.22
15	61.95	2.98
16	52.00	2.68
17	44.14	2.52
18	37.75	2.29
19	32.46	2.08
20	27.89	1.89
21	24.09	1.76
22	20.90	1.63
23	18.22	1.54
24	15.93	1.49
25	13.89	1.36
26	12.18	1.30
27	10.75	1.21
28	9.520	1.13
29	8.500	1.06
30	7.348	0.956
31	6.503	0.917
32	5.895	0.873
33	5.256	0.834
34	4.808	0.784
35	4.212	0.726
36	3.837	0.707
37	3.478	0.676
38	3.147	0.637
39	2.789	0.591
40	2.524	0.550
41	2.299	0.516
42	2.143	0.502
43	1.927	0.478
44	1.743	0.465
45	1.577	0.433
46	1.459	0.413
47	1.328	0.390
48	1.208	0.361
49	1.112	0.341
50	1.021	0.338

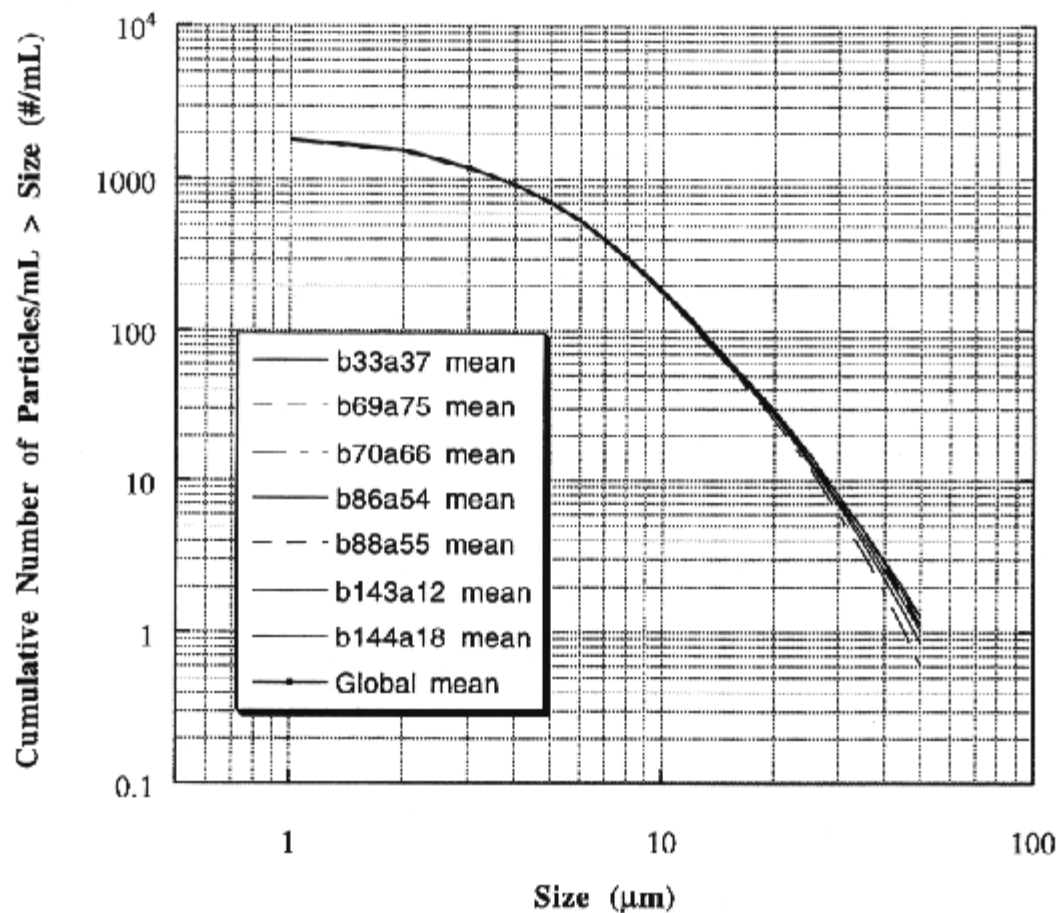


Figure 1. Cumulative Number of Particles per mL Greater than the Specified Size for eight bottles of RM 8631 Medium Test Dust (MTD) Measured by an Optical Particle Counter Calibrated to ISO 4402:1991.

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at Phone: (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet <http://ts.nist.gov/srm>.