National Institute of Standards & Technology

Report of Investigation

Reference Material 8631a

Medium Test Dust (MTD)

A unit of Reference Material (RM) 8631a, an ISO Medium Test Dust (MTD) [1], consists of 20 g of a natural mineral dust that is heterogeneous in composition and polydisperse with respect to size. RM 8631a is intended to be used as a secondary material for calibrating particle sizing instruments, especially optical particle counters, when used in conjunction with either of two published standard methods. RM 8631a can be used in conjunction with either the National Fluid Power Association method (NFPA) method NFPA/T2.9.11 R1-1998 "Hydraulic Fluid Power - Calibration of Liquid Automatic Particle Counters" [2] or the International Standards Organization method ISO 11171:1999 "Hydraulic Fluid Power - Calibration of Liquid Automatic Particle Counters" [3]. Using either of these two methods and following the Instructions for Use described below, the particle concentration of RM 8631a at each diameter will be determined by calibration against NIST Standard Reference Material 2806a, Medium Test Dust (MTD) in Hydraulic Fluid.

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.

Maintenance of RM Reference Values: NIST will monitor representative samples from this RM lot over the period of its validity. If substantive technical changes occur that affect the reference values before the expiration of this report, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Source of the Material[1]: This material was manufactured and donated by Powder Technologies, Inc., Burnsville, MN. RM 8631a is a derivative of Arizona Road Dust (ISO Medium Dust also known as PTI 5-80 Test Dust 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.

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 riffling, flat pancake sampling, or cone and quartering [4-6]. These sampling procedures require the entire bottle to be utilized in the reduction to arrive at a split aliquot for analysis.

Preparation and initial particle counting of hydraulic oil suspensions of RM 8631a were accomplished by the Institut de la Filtration et des Techniques Séparatives Liquide-Solide (IFTS), 3, Rue Marcel Pagnol, 47510 Foulayronnes, France.

The overall direction and coordination of the technical work required for this project and analysis of homogeneity was provided by R.A. Fletcher of the NIST Surface and Microanalysis Science Division and the NIST Chemical Science and Technology Laboratory.

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[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 identified are necessarily the best available for the purpose.

Experimental sampling design and statistical analysis of the data were provided by J.J. Filliben of the NIST Information Technology Laboratory.

Support aspects involved in the issuance of this RM were coordinated through the NIST Measurement Services Division.

Homogeneity Test: NIST tested the bottle-to-bottle particle number size homogeneity by examining selected bottles of the RM from the four quadrants of the spin riffler sampling wheel. Two adjacent bottles in each quadrant were chosen to facilitate decoupling the instrument measurement error from the actual variation in the 20 g vials.

The medium test dust was accurately weighed and the material was suspended in clean MIL-H-5606 hydraulic oil using a mixing test stand to produce suspensions of known concentration at a nominal 2.28 mg/L by the IFTS. The material is suspended according to ISO 11171 requiring 30 bottles of suspension traceable to each bottle of dry medium test dust. IFTS used a PAMAS optical particle extinction sensor calibrated using SRM 2806a. Relative measurements to determine the particle size distribution were made by NIST using an HIAC/ROYCO optical particle counter (HR-LD 600) equipped with a light extinction sensor calibrated using SRM 2806a. There were 143 valid analyses of the suspended MTD material in hydraulic oil.

The mean values for the size distribution derived for all 8 bottles of RM 8631a samples for which there were 143 total measurements are presented in Table 1. Table 1 shows the differential particle size defined by the ISO 11171:1999 calibration (SRM 2806a), the mean number of particles counted per milliliter of fluid, the standard uncertainty, the expanded uncertainty and the uncertainty in the mean for the example NIST characterization experiment. The mean value calculated across all 8 bottles of RM 8631a is plotted in Figure 1 showing the relative variation in the differential size distribution. Table 2 gives the standard deviation found for each bottle of dry MTD.

The purpose of RM 8631a is to be a source of MTD to make secondary standard MTD- hydraulic oil suspensions that in turn can be made traceable to NIST by the user through comparison with SRM 2806a. ISO 11171:1999 places no restriction on the source of MTD to make the secondary standards. Although the particle size analysis indicates that the 8 bottles analyzed are not statistically identical, they have size distributions that are nearly the same as evidenced by the low standard error in the means as shown in Table 1. The standard uncertainties in Table 1 compare favorably with the standard deviations (uncertainties) obtained for dry MTD bottle analysis shown in Table 2. The values in Table 2 are anywhere from 25 % to 200 % of the global standard uncertainties. At this time, it is impossible to tell whether the differences are due to the spin riffle process or due to the dry dust sampling, hydraulic oil suspension preparation or the instrumental analysis performed with the optical particle sensor.

Table 1: Mean Values of Particles per mL Suspension Averaged over all Samples Measured by an Optical Particle Counter Calibrated to ISO 11171:1999.

Size[(a)] (µm)	Mean Concentration (particles/mL)	Standard Uncertainty (particles/mL)	Expanded Uncertainty[(b)] (particles/mL)	Standard Uncertainty in the Mean (n=143) (particles/mL)
4	1609	204	408	17.1
5	501	20.5	40.9	1.71
6	227	8.65	17.3	0.723
7	122	3.80	7.61	0.318
8	75.1	2.76	5.52	0.231
9	50.9	2.41	4.82	0.201
10	26.7	1.62	3.24	0.135
12	9.09	0.652	1.30	0.055
14	3.73	0.245	0.489	0.020
17	1.46	0.136	0.273	0.011
20	0.639	0.087	0.174	0.007
22	0.383	0.076	0.152	0.006
25	0.183	0.045	0.091	0.004
30	0.070	0.028	0.056	0.002
35	0.026	0.018	0.037	0.002
40	0.015	0.014	0.027	0.001

(a) Particle size obtained from ISO 11171:1999.

(b) Following NIST Technical Note 1297 [8], the uncertainties correspond to the expanded uncertainties in the particle concentration which are twice the standard uncertainties (NIST specification of $k = 2$). The standard uncertainty is the square root of the sum-of-the-squares of the individual standard deviations derived from the measurement process. Level $k = 2$ defines an interval with a confidence of approximately 95%.

Table 2. Standard Deviations (uncertainty) Determined for each Bottle of Dry MTD (n = 18). The Particle Size is Obtained from SRM 2806a.

Size (µm)	b1b (particles/mL)	b36b (particles/mL)	b37b (particles/mL)	b73b (particles/mL)	b74b (particles/mL)	b110b (particles/mL)	b111b (particles/mL)	b147b (particles/mL)
4	91.4	421.9	160.5	61.1	94.5	114.2	128.7	149.1
5	22.7	25.7	7.2	12.6	6.0	6.7	16.4	23.3
6	7.2	5.0	4.2	5.4	3.2	3.6	7.4	10.4
7	1.2	2.3	2.9	2.9	1.6	1.4	3.5	3.5
8	1.6	1.8	1.8	1.7	1.1	1.0	1.9	1.7
9	1.6	1.5	1.3	1.7	0.776	0.738	1.8	1.4
10	1.0	1.0	0.605	1.0	0.645	0.507	1.4	0.953
12	0.478	0.474	0.388	0.475	0.365	0.431	0.561	0.527
14	0.193	0.228	0.174	0.122	0.208	0.161	0.335	0.213
17	0.115	0.134	0.102	0.122	0.137	0.099	0.144	0.145
20	0.074	0.089	0.101	0.079	0.086	0.079	0.081	0.090
22	0.069	0.060	0.063	0.063	0.068	0.072	0.115	0.079
25	0.046	0.054	0.052	0.032	0.033	0.032	0.059	0.043
30	0.034	0.025	0.027	0.035	0.021	0.032	0.025	0.025
35	0.015	0.015	0.016	0.017	0.014	0.019	0.025	0.023
40	0.014	0.013	0.015	0.012	0.012	0.016	0.014	0.014

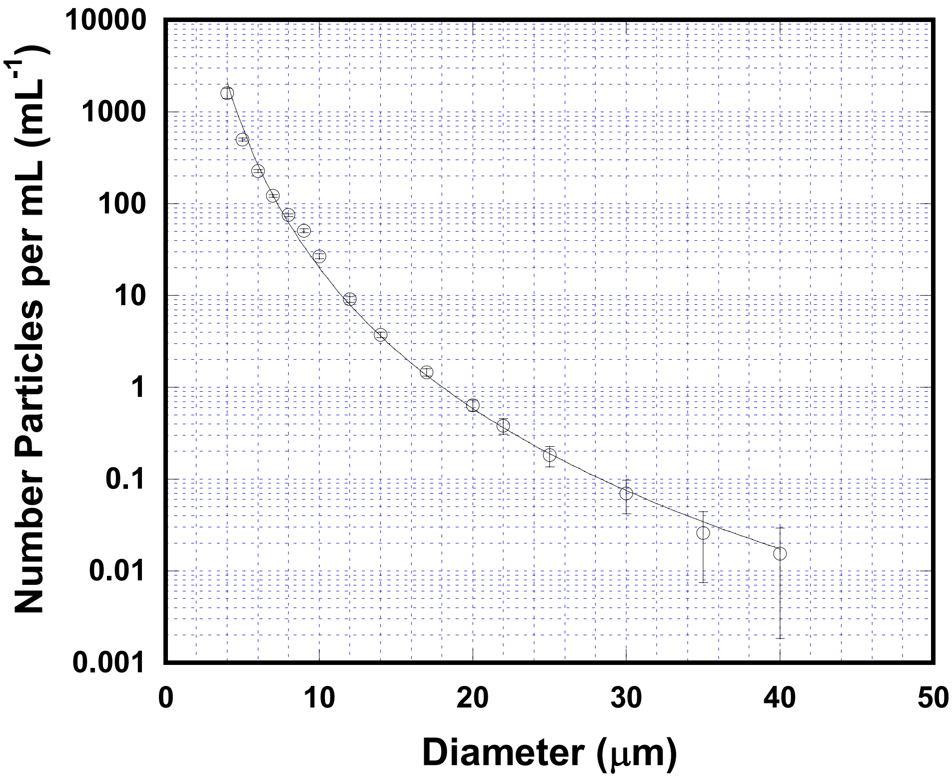

Figure 1. Differential mean particle count per mL of hydraulic fluid for 8 different bottles of RM 8631a MTD measured by an optical particle counter calibrated to ISO 11171:1999. The curve is a power law fit to the data. The data points represent the global mean for all data. The uncertainty is expressed as the standard deviation (n = 143). The variation for the small particles is too small to be visualized on the plot.

REFERENCES

[1] ISO 12103; *Test Dust For Filter Evaluation Part I - Arizona Test Dust*; International Organization for Standardization: Geneve, Switzerland; Case Postale 56. CH 1211

[2] NFPA T2.9.11 R1-1999. *Hydraulic Fluid Power – Method for Determining the Particulate Count of an Oil Sample (using Liquid Automatic Counters)*; National Fluid Power Association, Milwaukee, WI. Revision of ANSI/(NFPA)T2.9.11-R2004

[3] ISO 11171: ISO/TC 131/SC 6 N 202; *Hydraulic Fluid Power - Calibration of Liquid Automatic Particle Counters*; International Organization for Standardization: Case Postale 56. CH-1211 Geneve, Switzerland (1999).

[4] ASTM C 702-93; *Standard Practice for Reducing Samples of Aggregate to Testing Size*.

[5] Allen, T.; *Particle Size Measurement*; Chapman and Hall, London, p. 23 (1974).

[6] Pitard, F.F.; *Pierre Gy's Sampling Theory and Sampling Practice*; CRC Press, Ann Arbor, pp. 240-241 (1995).

[7] Croarkin, M.C.; Tobias, P.; Guthrie, W.F.; Hembree, B.; Filliben, J.J.; Heckert, N.A.; Prinz, J.; Zey, C.; *NIST/SEMATECH e-Handbook of Statistical Methods*; NIST Handbook 151; U.S. Printing Office: Washington, DC (2003); available at http://www.itl.nist.gov/div898/handbook/.

[8] ISO; G*uide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); 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://physics.nist.gov/Pubs/.

Users of this RM should ensure that the report in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.