National Institute of Standards & Technology

Report of Investigation

Reference Material 8631b

Medium Test Dust (MTD)

Reference Material (RM) 8631b is intended to be used as a secondary material for calibrating particle sizing instruments, especially optical particle counters, when used in conjunction with published standard methods [1,2]. A unit of RM 8631b consists of 20 g of a natural mineral dust that is heterogeneous in composition and polydisperse with respect to size.

RM 8631b can be used in conjunction with the International Standards Organization method ISO 11171:2016 *Hydraulic Fluid Power - Calibration of Liquid Automatic Particle Counters* [2]. Using this method and following the "Instructions for Use" described in this report, the particle concentration of RM 8631b at each diameter will be determined by calibration against NIST Standard Reference Material 2806x, *Medium Test Dust (MTD) in Hydraulic Fluid*.

Information Values: Information values for RM 8631b are shown in Table 1, Table 2, and Figure 1. Information values cannot be used to establish metrological traceability.

NOTICE 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.

Expiration of Material: The material comprising this RM should remain stable indefinitely. The reference material will 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: NIST will monitor this RM over the period of its validity. If substantive technical changes occur that affect this report, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of the technical work required for this project and analysis of homogeneity was provided by R.A. Fletcher and N.W M. Ritchie of the NIST Materials Measurement Science Division.

Experimental sampling design and statistical analysis of the data were provided by J.J. Filliben, of the NIST Statistical Engineering Division.

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

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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 sub-samples from the RM bottle. To sub-sample, follow an accepted procedure including spin riffling, 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. The purpose of RM 8631b is to be a source of MTD to make secondary standard MTD-hydraulic fluid suspensions that in turn can be made traceable to NIST SRM 2806 by the user through comparison with SRM 2806x. ISO 11171:2016 does not place a restriction on the source of MTD to make the secondary standards. However, the international fluid power community desires a repository of ISO MTD to be made available. This MTD has the property that it was taken from the same batch number 4390C as the MTD used to make SRM 2806x. Companies that make secondary standards for sale will find it beneficial to use RM 8631b MTD because the product closely matches the size distribution for SRM 2806x. Note: General specification 2806x refers to future 2806 issues, i.e., 2806d, 2806e, etc.

SOURCE, PREPARATION, AND ANALYSIS(1)

Source: This material was manufactured and donated by Powder Technologies, Inc. (Burnsville, MN). RM 8631b 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 approximately 20 g aliquots by Laboratory Quality Services International (South Holland, IL).

Preparation and Analysis: The spin riffling process resulted in 147 bottles of 20 g samples for distribution. NIST tested the bottle-to-bottle particle number size homogeneity by examining 8 selected bottles of the RM from the four quadrants of the spin riffler sampling wheel. Two adjacent bottles in each quadrant were chosen to analyze with the purpose of decoupling the instrument measurement error from the actual variation in the 20 g vials. The bottles chosen for analysis are B12C, B13C, B42C, B43C, B77C, B78C, B135C and B136C. A NIST spin riffle splitter was used to further split the 20 g sample down to useable mass; this riffler divides the particle sample into 16 sub-samples that are considered to be identical. Some of those sub-samples are combined and further divided using the riffler. Each 20 g bottle of RM 8631b was riffle split down to samples of approximately 0.015 g. The mass of each final MTD aliquot is variable so each size distribution is scaled to the respective MTD mass of the respective aliquot. For each bottle of MTD, 5 separate ca. 0.015 g aliquots were separately analyzed, and results combined to find mean and standard deviation for each original bottle (for example, B77C). The position for each split was randomized but balanced so that the splitting would not be biased due to some unexpected mechanical action of the riffle splitter. Every MTD sub-sample was weighed for mass determination and the mass was used to scale the particle count measurements.

Five sub-splits of MTD from each 20 g bottle, with mass approximately 0.015 g, were separately analyzed. Following the procedure outlined in British Standard ISO 12103-1:1997 [6] each ca. 0.015 g MTD split was suspended in 11 mL of filtered (0.40 μm pore) deionized water using 1 drop of a non-polar dispersant (A1 dispersant, Beckman Coulter). The suspension was mixed on a mechanical mixer and sonicated for 10 s and then mixed once again. Two mL of suspension were immediately drawn by a calibrated pipette and injected into 200 mL of ISOTON II (Beckman Coulter). The new diluted suspension was stirred with a mechanical stirring device continuously and analyzed using a Coulter Multisizer IIe (Beckman Coulter Counter), an electrozone particle analyzer. The Coulter Counter was equipped with a 100 μm orifice. From 3 to 5 repeat analysis were done on each suspension, (i.e., on each sub-split for each bottle). One analysis consisted of counting and sizing the particles from 2 μm to 60 μm in 2 mL of resultant suspension formed in the 202 mL ISOTON II particle suspension. Blanks were taken to insure clean solutions and low contamination.

(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.

Table 1 shows the mean values for the cumulative number of particles greater than specified diameter per 1 mg of MTD for each bottle analyzed by the Coulter Counter. The means are composed of 5 separate independent dust sub-samples collected randomly from the final 16 unit-split off the riffle splitter. For example, for B78C there were sub-samples B78C-1, B78C-2, B78C-5, B78C-10, and B78C-13 where the trailing integer corresponds to the 1 to 16 location on the splitter wheel. For each sub-sample, e.g., B78C-10, there are 3 to 5 analyses from the suspension as described above. The result is that for each bottle there have been 15 to 25 measurements of the particle size distribution. Since the number of particles in a constant volume of suspension is directly related to the mass concentration of the suspension, each suspension was scaled by the mass of the MTD used to make up the sample. All dilutions were described above; each mass aliquot of MDT was suspended in the same volume of water.

Table 1. Information Mean Values (n =15 to 25) for the Cumulative Number of Particles per Milligram for Each Bottle Analyzed by the Coulter Counter

Diameter	Sample B12C	Sample B13C	Sample B42C	Sample B43C	Sample B77C	Sample B78C	Sample B135C	Sample B136C
(µm)	(number particles/mg)							
2	3.575×10^6	3.708×10^6	3.485×10^6	3.527×10^6	3.435×10^6	3.663×10^6	3.620×10^6	3.619×10^6
3	1.741×10^6	1.813×10^6	1.716×10^6	1.745×10^6	1.682×10^6	1.803×10^6	1.761×10^6	1.777×10^6
4	1.014×10^6	1.063×10^6	1.006×10^6	1.024×10^6	9.852×10^5	1.047×10^6	1.023×10^6	1.033×10^6
5	6.112×10^5	6.382×10^5	6.036×10^5	6.163×10^5	5.910×10^5	6.305×10^5	6.130×10^5	6.258×10^5
6	3.723×10^5	3.861×10^5	3.659×10^5	3.738×10^5	3.581×10^5	3.817×10^5	3.714×10^5	3.813×10^5
7	2.341×10^5	2.425×10^5	2.296×10^5	2.350×10^5	2.248×10^5	2.393×10^5	2.317×10^5	2.402×10^5
8	1.538×10^5	1.588×10^5	1.508×10^5	1.547×10^5	1.476×10^5	1.575×10^5	1.516×10^5	1.586×10^5
9	1.065×10^5	1.100×10^5	1.051×10^5	1.077×10^5	1.021×10^5	1.093×10^5	1.051×10^5	1.102×10^5
10	7.723×10^4	7.944×10^4	7.645×10^4	7.821×10^4	7.350×10^4	7.884×10^4	7.591×10^4	8.014×10^4
12	4.440×10^4	4.556×10^4	4.457×10^4	4.545×10^4	4.241×10^4	4.553×10^4	4.387×10^4	4.680×10^4
14	2.774×10^4	2.825×10^4	2.783×10^4	2.841×10^4	2.591×10^4	2.820×10^4	2.731×10^4	2.932×10^4
17	1.448×10^4	1.473×10^4	1.480×10^4	1.518×10^4	1.367×10^4	1.475×10^4	1.432×10^4	1.543×10^4
20	7.949×10^3	7.783×10^3	8.077×10^3	8.377×10^3	7.421×10^3	7.855×10^3	7.649×10^3	8.588×10^3
22	5.338×10^3	5.166×10^3	5.527×10^3	5.716×10^3	4.888×10^3	5.283×10^3	5.127×10^3	5.911×10^3
25	3.011×10^3	2.916×10^3	3.166×10^3	3.244×10^3	2.894×10^3	2.905×10^3	2.911×10^3	3.355×10^3
30	1.144×10^3	1.189×10^3	1.328×10^3	1.411×10^3	1.172×10^3	1.161×10^3	1.155×10^3	1.417×10^3
35	5.333×10^2	5.444×10^2	5.833×10^2	6.055×10^2	5.222×10^2	4.833×10^2	4.555×10^2	6.111×10^2
40	2.555×10^2	2.666×10^2	2.611×10^2	2.611×10^2	2.500×10^2	2.222×10^2	2.278×10^2	2.833×10^2

Table 2 shows the global mean found from averaging all 8 bottle results, standard deviation and standard uncertainty for the number of particles per 1 mg. The values correspond to the measured cumulative number of particles greater than specified diameter per 1 mg of MTD. Based on the suspension dilution, mass of MTD and the 2 mL Coulter sample size, we calculate the mass of the dust analyzed by the Coulter Counter to be in the range of 18 µg to 53 µg.

Table 2. Information Average Values Over All bottles (Global Average, n = 8) of Cumulative Number of Particles Per Milligram Larger Than Specified Diameter

Diameter	Global Mean	Global Standard Deviation	Global Standard Uncertainty, *k* = 2[(a)]	Relative Standard Uncertainty, *k* = 2[(a)]
(µm)	(number particles/mg)	(number particles/mg)	(number particles/mg)	(%)
2	3.579×10^6	9.201×10^4	6.506×10^4	1.8
3	1.755×10^6	4.378×10^4	3.096×10^4	1.8
4	1.025×10^6	2.419×10^4	1.710×10^4	1.7
5	6.162×10^5	1.518×10^4	1.073×10^4	1.7
6	3.738×10^5	9.158×10^3	6.476×10^3	1.7
7	2.347×10^5	5.927×10^3	4.191×10^3	1.8
8	1.542×10^5	4.024×10^3	2.845×10^3	1.9
9	1.070×10^5	2.838×10^3	2.007×10^3	1.9
10	7.747×10^4	2.162×10^3	1.529×10^3	2.0
12	4.482×10^4	1.326×10^3	9.380×10^2	2.1
14	2.787×10^4	9.874×10^2	6.982×10^2	2.5
17	1.467×10^4	5.380×10^2	3.804×10^2	2.6
20	7.962×10^3	3.801×10^2	2.687×10^2	3.4
22	5.370×10^3	3.333×10^2	2.357×10^2	4.4
25	3.050×10^3	1.806×10^2	1.277×10^2	4.2
30	1.247×10^3	1.180×10^2	8.343×10^1	6.7
35	5.423×10^2	5.594×10^1	3.956×10^1	7.3
40	2.534×10^2	2.012×10^1	1.423×10^1	5.6

[(a)] k = 2 is interpreted as approximately the 95 % confidence level.

Homogeneity: There appears to be no systematic bias in the MTD size distributions as a function of the dry dust bottle position on the riffle table. Table 2 shows a relative standard uncertainty (at k = 2 level) that is quite small – from 1.7 % to 7.3% for most particle sizes. RM 8631b will provide a good secondary standard material for the fluid power community. The variation found in the new MTD is small and thus the batch bottle-to-bottle variation of MTD is sufficiently homogeneous and fit for purpose.

Information values regarding the homogeneity of the 8 bottles are shown in Figure l. The horizontal axis is the 30 particle diameters. The vertical axis is the normalized values for each bottle at each diameter. For each diameter, the normalization is done by computing the mean M and standard deviation SD across all 8 bottles. The value X is taken as the mean over the n = 5 independent particle suspension measurements within a bottle. The vertical axis normalized "score" is then computed as Z = (X-M)/SD.

All 8 of the bottles have their mean within 1.2 standard deviations of the 8-bottle mean for each (and every) diameter. This indicates that from a practical point of view, the 8 bottles (and inferentially the batch) are deemed to be sufficiently homogeneous for the metrological purpose.

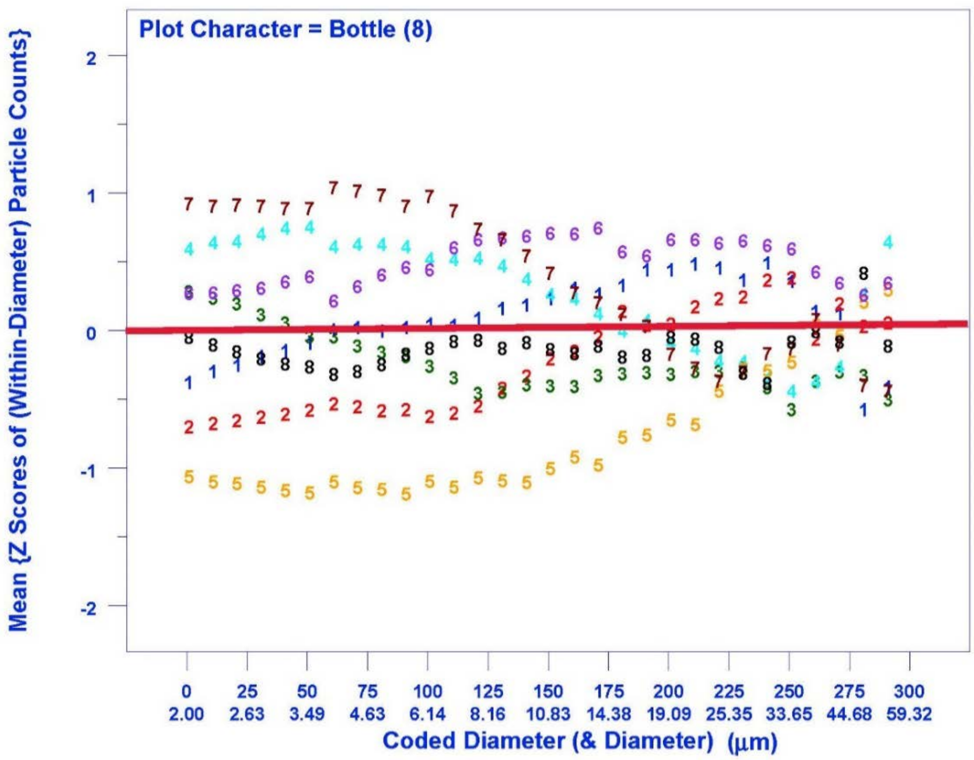

Figure 1. Z plot where Z = (X-M)/SD, X is mean of 5 independent suspensions, M is the global mean of bottles 1 to 8 and SD is the respective global standard deviation. Values run from approximately 2 µm to 59.32 µm or from 0 to 300 for the coded diameters. The Z-values correspond to each bottle as follows (1) B43C, (2) B42C, (3) B135C, (4) B78C, (5) B77C, (6) B136C, (7) B13C, and (8) B12C.

REFERENCES

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