National Institute of Standards & Technology

Report of Investigation

Reference Material 8632a

Ultra Fine Test Dust (UFTD)

Reference Material (RM) 8632a, Ultra Fine Test Dust (UFTD) [1], consists of 20 g of a natural mineral dust (batch 13458D) that is heterogeneous in composition and polydisperse with respect to particle size. RM 8632a is intended to be used as a secondary test material for verifying particle sizing instruments, especially automatic optical particle counters, when used in conjunction with the published standard method, International Standards Organization method ISO 11171:2020 *Hydraulic Fluid Power — Calibration of Liquid Automatic Particle Counters* [2] Table A.1. Using ISO 11171:2020 and following the Instructions for Use described below, the cumulative particle concentration of RM 8632a greater than each diameter will be used to assess the automatic particle counter validity.

Expiration of Value Assignment: RM 8632a is valid indefinitely, within the measurement uncertainties specified, provided the RM is handled and stored in accordance with instructions given in this Report of Investigation (see "Instructions for Handling, Storage, and Use"). Periodic validation of this RM is not required. This report is nullified if the RM is damaged, contaminated, or otherwise modified.

Maintenance of RM: The values in this report are for informational purposes only. Before using any of the values derived from this material, users should obtain the most recent version of this documentation, available free of charge through the https://www.nist.gov/srm website.

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.

Statistical analysis of the data was provided by J.J. Filliben of the NIST Statistical Engineering Division.

B. Verdegan, Cummins Filtration (Stoughton, WI) and 11171 Solutions (Stoughton, WI), coordinated the interlaboratory study (ILS) effort that provided the measurements results for RM 8632a. The ILS was made possible by 13 laboratories and 10 companies, located globally, voluntarily providing analysis and reports of the candidate RM 8632a: ARGO-HYTOS GMBH (Kraichtal, Germany); Aviation Industry Metrology and Test Science Technology Co., Ltd. (Xinxiang City, China); Beckman Coulter (Grants Pass, OR); Cummins Filtration; Donaldson (Bloomington, MN); Fluid Technologies, Inc. (Stillwater OK); Institut de la Filtration et des Techniques Separatives (IFTS; Foulayronnes, France); PAMAS (Rutesheim, Germany); Parker Hannifin (Kearney, NE); Southwest Research Institute (San Antonio, TX), and Stanhope-Seta, Ltd (Chertsey, UK).

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

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INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

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

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 [3–5]. These sampling procedures require the entire bottle to be utilized in the reduction to arrive at a split aliquot for analysis.

PREPARATION AND ANALYSIS(1)

This material was manufactured by Powder Technologies, Inc., Arden Hills, MN. RM 8632a is a derivative of Arizona Road Dust that was taken from the production lot of Ultra Fine Test Dust No. 13458D. Approximately 7 kg of material was spin-riffled, bottled, and sealed in containers holding 20 g aliquots by Laboratory Quality Services International, South Holland, IL.

Homogeneity Test: The spin riffling process resulted ideally in 323 bottles of 20 g identical samples for distribution. An ILS was conducted to determine the values under actual measurement conditions prescribed in ISO 11171:2020 [2] associated with SRM 2806d and with RM 8632a. Homogeneity was determined from measuring 13 selected bottles of the RM distributed across the spin riffler sampling wheel. The bottles tested were the following: 007, 019, 071, 090, 123, 146, 177, 186, 227, 238, 265, 293, and 308. These bottles were sent to 13 individual laboratories participating in an ILS for SRM 2806d and RM 8632a (see ISO/TR 4813:2020 Hydraulic fluid power — Background, impact and use of ISO 11171:2020 on particle count and filter test data). Each lab was tasked with measuring the cumulative particle size distribution for three 1 mg/L RM 8632a particle suspensions made by each lab from dry RM 8632a and particle free MIL PRF 5606 hydraulic fluid. The labs were to use automatic particle counters calibrated to SRM 2806b through a secondary reference material (see Appendix A) to measure the cumulative particle size distribution. The suspensions were to be diluted according to the procedures outlined in ISO 11171:2020 [2] where necessary. The concentrations presented in Table 1 are the median of the mean and mean of the mean suspension cumulative particle concentrations of 1 mg/L found by the participating labs.

Reference Values: Reference values for RM 8632a are reported in Table 1 and are the median of the means and the mean of the means of the cumulative particle concentration. A NIST Reference value is a non-certified value that is the present best estimate based on available data; however, the value does not meet the NIST criteria for certification and is provided with an associated uncertainty that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [7]. The value is the unweighted mean or median of results obtained at collaborating laboratories. The expanded uncertainty, U, is an expanded uncertainty about the mean, with listed coverage factor, k [6,7]. Results derived from the use of this value are considered by NIST to be traceable only to the value itself. The ($k = 2$) uncertainty was determined using Bootstrap calculations and approximately corresponds to 95 % confidence interval.

(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. RM 8632a ILS results presenting reference values for the median of the means and the mean of the means of the cumulative particle concentration with their associated expanded ($k = 2$) uncertainty, U [6,7]. The number of measurements is shown in the last column.

Diameter [µm(c)][(a)]	Median of Mean Cumulative Particle Concentration [part./mL][(b)]	Expanded Uncertainty Median of the Mean Cumulative Particle Concentration ($k = 2$) [part./mL]	Relative Expanded Uncertainty in Median Mean ($k = 2$) [%]	Mean of the Mean Cumulative Particle Concentration [part./mL]	Expanded Uncertainty Mean of the Mean Cumulative Particle Concentration ($k = 2$) [part./mL]	Relative Expanded Uncertainty in Mean of Mean ($k = 2$) [%]	Number of Measurements
-	-	-	-	-	-	-	-
2	20 213.1	3 268.9	16.2	19 377.7	2 562.3	13.2	3
3	10 699.3	734.9	6.9	10 861.1	608.3	5.6	13
4	6 298.9	372.9	5.9	6 544.9	360.6	5.5	17
5	3 475.2	154.2	4.4	3 591.9	250.2	7.0	17
6	1 630.6	123.3	7.6	1 762	167.8	9.5	17
7	769.8	62.2	8.1	845.6	92.4	10.9	16
8	379.1	52.7	13.9	426.1	53.9	12.6	16
9	196.6	54.6	27.8	219.2	35.5	16.2	14
10	88.9	37.3	42.0	113.5	20.8	18.3	17
11	43.4	27.3	62.8	63	18.2	28.9	13
12	22.6	10.8	47.6	34.2	12	35.1	15
13	12.3	12.2	98.6	23.4	11	47.0	13
14	8.3	6.7	80.7	15.6	7.6	48.7	16
15	5.2	4.1	78.2	11.3	7	61.9	15

(a) The particle diameter in micrometer(c) [µm(c)] is listed in column 1. Micrometer(c) [µm(c)] is a unit assigned to particle diameters reported in the original SRM 2806 and SRM 2806b material, both materials traceable to the NIST Line Scale Interferometer, and provides continuity back to the original material and associated measurements. The relationship of 0.898 µm = 1 µm(c) has been adopted by ISO TC131 SC6 WG1 [see ISO Technical Report 22681 (2019)] [8].
(b) The particle concentration is listed in particles per milliliter (part./mL).

The values present in Table 2 and reference 2 (ISO 11171:2020 [2], Table A.1 "Particle size distribution for sensor performance verification") were derived from the $k = 3$ (approximately 3 sigma) of the log transformed uncertainty associated with the median of the mean ILS measured values. All of the ILS results fell between these upper and lower bounds.

Table 2. Values in ISO 11171:2020 [2] Table A.1 showing the lower and upper bound cumulative particle concentration greater than diameter permitted in the ISO standard [2].

Particle size [µm(c)]	Median particle concentration greater than indicated size for a 1 mg/L sample of RM 8632a (particles/mL)	Particle concentration (particles/mL) greater than indicated size for a 1 mg/L sample of RM 8632a	
		greater than or equal to	less than
2	20 210	14 180	28 820
3	10 700	7 928	14 440
4	6 300	4 553	8 713
5	3 475	2 330	5 183
6	1 631	938.2	2 834
7	769.8	413.6	1 433
8	379.1	179.7	799.5
9	196.2	76.57	502.6
10	88.90	27.89	283.4

Summary: The value and relevance of RM 8632a to the user community is that it provides a universal single source of UFTD to test automatic particle counters according to ISO 11171:2020 [2]. ISO 11171:2020 requires the source of UFTD to be NIST RM 8632a (Table A.1, Annex A in ISO 11171:2020) [2]. NIST's objective as per request of the Fluid Power Community is to provide this material for the standard method. The values presented in this report of investigation will be used as upper and lower bounds in the new version of ISO 11171:2020 [2] as required automatic particle counter responses for ISO 11171:2020 Table A.1 Annex A [2], "Particle size distribution for sensor performance verification."

There appears to be no systematic bias in the UFTD size distributions as a function of the dry dust bottle position on the riffle table. Table 2 provides a range of acceptability presented in ISO 11171:2020 [2] and is the median of the mean log transform ± 3 standard deviations ($k = 3$).

The bottle-to-bottle variability should be low based on the production process that used a riffle splitter to make the 323 individual 20 g samples. The homogeneity was not rigorously verified in the ILS because of general lack of individual lab analysis of multiple bottles. What was tested was the measurement variability for a set of very knowledgeable labs analyzing the candidate RM 8632a material. Each lab, aside from one lab, analyzed only a single bottle of RM 8632a. However, each lab used the procedure they would use following ISO 11171:2020 [2] making 3 suspensions from a single bottle of UFTD. Given the level of reproducibility found for 10 laboratories, 17 data sets measuring the RM 8632a UFTD in hydraulic fluid as shown in Table 1, under multiple lab, real test conditions, the new RM UFTD provides realistic values with sufficient low variability to serve for its necessary application as a test dust given the broad acceptance range in Table 2 (Table A.1, Annex A "Particle size distribution for sensor performance verification", ISO 11171:2020 [2]).

REFERENCES

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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; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm.

Appendix A

Table A1. Summary of Laboratory Participation[(a,b)]

1 Data Set	2 ILS lab ID	3 Actual Lab ID	4 Instrument Used	5 Instrument Technology	6 Bottle # of RM 8632a
1	1a	1	1	E	308
2	2a	2	2	E	90
3	3a	3	1	E	7
	4a	3	1	S	19
4	3b	3	1	E	19
5	4b	3	1	E	19
	5a	4	1	E	318
6	6a	5	1	E	238
7	7a	6	3	E	186
8	8a	7	2	E	293
9	9a	8	2	E	71
10	9b	8	2	E	177
	10a	9	1	E	201
	11a	10	1	E	213
11	12a	11	1	E	227
12	13a	11	1	S	123
13	14a	12	1	E	146
14	15a	13	1	E	265
15	16a	5	1	S	238
16	17a	7	2	E	293
17	18a	6	3	E	186

(a) Column Definitions: **Column 1:** Data set number. **Column 2:** Original ILS assigned id number. **Column 3:** The true lab id assigned to each of the 13 labs (10 labs) for the analysis. **Column 4:** Instrument used by the participant (1 – PAMAS, 2 – Beckman Coulter, 3 – Stanhope-Seta). **Column 5:** technology used by the instrument either extinction (E) or light scattering (S). **Column 6:** **T**he bottle number of RM 8632a used in the ILS.

(b) Gray shading indicates that the lab's data was missing from the analysis.

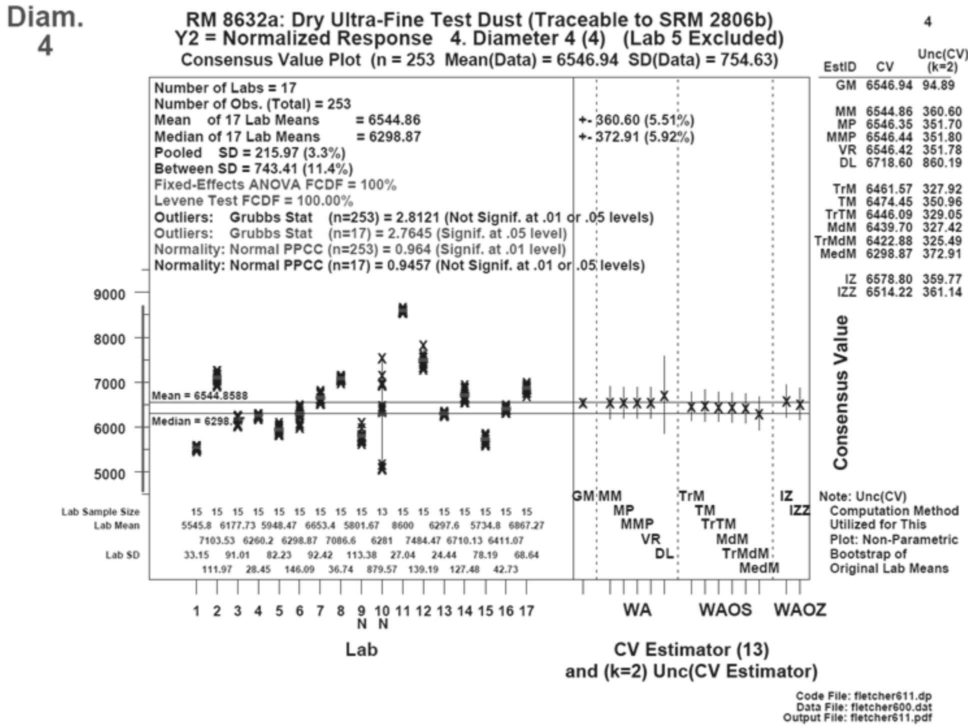

Figure A1. Plot showing 17 data sets of cumulative particle concentration > diameter for the suspensions at 4 µm(c). Each cluster of points represents 15 measurements. The plot shows graphically the within-lab variation and across-lab agreement.

The consensus values were found using DATAPLOT [9] code to calculate the median of the mean and the mean of the mean values as well as other statistics. A plot for just the 4 µm(c) results is shown in Figure A1. Median of the mean (MedM) and mean of the mean (MM) are presented in the columns to the right of the plot as well as the uncertainty (Unc) associated with these values, determined by a Bootstrap statistical method.

Table A2. Complete data set provided by 10 contributing laboratories providing 17 sets of measurements. The identifiers above the columns, (for example, 1a, 2a, etc.) are the original laboratory assigned identities (see Table A1). Several labs performed analysis on multiple instruments leading to 17 data sets identified in each column by Lab 1, Lab 2, etc. Each value is the mean (n = 15) of the cumulative particle concentration in particles/mL > diameter determined from the consensus calculation shown in Figure A1 carried out for all relevant diameters. The mean of the mean and median of the mean are given at the far right with n = the number of measurements.

	1a	2a	3a	3b	4b	6a	7a	8a	9a(N)	9b(N)	12a	13a	14a	15a	16a	17a	18a			
Diam	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	Lab 7	Lab 8	Lab 9	Lab 10	Lab 11	Lab 12	Lab 13	Lab 14	Lab 15	Lab 16	Lab 17	n	Mean	Median
1.5															22656.07			1	22656.07	22656.07
2					20213.07							21057.73			16862.20			3	19377.67	20213.07
3	10299.40	12688.13	10699.27	10928.07	10266.93	10743.13		12572.07	9859.80	10635.15		12032.73	11692.20		9393.93	9383.33		13	10861.09	10699.27
4	5545.80	7103.53	6177.73	6260.20	5948.47	6298.87	6653.40	7086.60	5801.67	6281.00	8600.00	7484.47	6297.60	6710.13	5734.80	6411.07	6867.27	17	6544.86	6298.87
5	2807.07	3852.20	3297.73	3308.53	3728.93	3357.20	3542.07	3584.87	3209.93	3510.00	4907.60	4723.00	3405.67	3475.20	3429.47	3290.60	3632.40	17	3591.91	3475.20
6	1214.67	1782.47	1541.00	1529.40	2338.20	1589.53	1630.60	1657.93	1593.60	1771.85	2406.93	2518.67	1596.40	1602.13	1897.33	1595.80	1687.40	17	1761.99	1630.60
7	541.67	847.00	749.93	742.47		809.93	746.53	759.07	806.40	915.85	1195.13	1234.00	761.07	759.33	1122.07	768.07	771.47	16	845.62	769.77
8	235.96	424.22	370.60	359.78		433.79	358.11	382.13	423.31	501.40	595.48	605.82	370.07	376.07	639.64	370.67	370.03	16	426.07	379.10
9	103.49	208.72				255.19	173.12	184.41	228.68	285.60	279.30	285.03	169.55	179.00	358.56	181.47	176.55	14	219.19	196.57
10	44.34	99.94	84.26	79.23	182.93	162.89	85.73	88.89	134.13	171.37	130.82	133.02	79.66	85.47	191.18	88.80	86.75	17	113.50	88.89
11	20.91					112.16	41.27	43.23	78.67	107.17	62.81	65.19	37.64	41.33	123.51	43.40	41.55	13	62.99	43.40
12	10.77		24.51	21.24		85.54	20.21	21.34	48.39	68.75	28.43	28.96	19.60	19.13	72.84	22.60	20.51	15	34.19	22.60
13	6.55					70.36	11.99	11.34	32.60	50.45	14.69	15.11	12.14	10.13	45.13	12.33	11.77	13	23.43	12.33
14	4.78		9.51	5.98	22.93	59.61	7.37	7.00	23.45	37.15	9.08	9.07	6.89	6.20	27.01	7.53	6.55	16	15.63	8.30
15	3.67		6.17	3.52		52.20	4.58	4.82	17.05	28.60	5.73	5.34	3.87	4.33	19.95	5.20	4.55	15	11.31	5.20

Secondary Calibration Material

The 13-lab ILS required a secondary calibration material because there was not sufficient SRM 2806b to serve as a direct calibrant for the study. Table A3 summarizes the properties of the secondary material and provides the total uncertainty associated with the secondary material. This uncertainty is propagated throughout the results of the ILS.

Table A3. The values and uncertainties of the Secondary Material used as calibrant in the ILS.

1 Projected Area Particle Diameter [µm(c)]	2 Median of Means of Cumulative Particle Concentration Secondary Calibration Material [part./mL]	3 Expanded Uncertainty of Median of Mean Cumulative Particle Concentration Secondary Calibration Material ($k = 2$) [part./mL]	4 Expanded Uncertainty in Projected Area Diameter Secondary Calibration Material ($k = 2$) [µm(c)]	5 Mean of Cumulative Particle Concentration SRM 2806b [part./mL]	6 Standard Uncertainty of Mean Cumulative Particle Concentration SRM 2806b ($k = 1$) [part./mL]	7 Expanded Uncertainty in Projected Area Diameter SRM 2806b (at 95 %) [µm]	8 Combined Expanded Uncertainty Cumulative Particle Conc. In Secondary Calibration Material ($k = 2$) [part./mL] [Q(3,6)][(a)]	9 $U^{(b)}$ Expanded Total Uncertainty in Projected Area Diameter In Secondary Calibration Material ($k = 2$) [µm(c)] [Q(4,7)][(c)]
1	-	-	-	80 755	1 318.7	0.26	-	-
2	41 069.18	175.68	0.0054	33 064	530.9	0.28	1 076.24	0.3
3	19 508.54	2 672.56	0.2043	17 714	305.2	0.29	2 741.38	0.4
4	12 016.34	976.21	0.1787	10 864	253.5	1.0	1 100.02	1.0
5	7 925.69	846.91	0.2521	6 681.2	127.6	1.0	884.52	1.0
6	5 296.74	752.44	0.3520	4 210.2	82.78	1.0	770.44	1.1
7	3 649.99	581.49	0.4252	2 852.3	61.18	1.1	594.22	1.2
8	2 561.59	264.13	0.2844	2 007	38.64	1.1	275.20	1.1
9	1 792.38	29.93	0.0467	1 476.4	27.9	1.1	63.32	1.1
10	1 280.08	79.35	0.1837	1 148.8	18	1.1	87.13	1.1
11	928.59	76.96	0.2593	857.22	13.61	2.0	81.63	2.0
12	686.39	56.89	0.2847	649.63	11.09	2.0	61.06	2.0
13	529.01	37.14	0.2743	500.66	10.12	2.0	42.30	2.0
14	415.55	28.28	0.2851	389.26	10.74	2.1	35.51	2.1
15	330.64	28.24	0.3764	299.96	8.79	2.1	33.26	2.2
16	265.49	26.83	0.4541	230.39	7.98	2.1	31.22	2.2
17	212.47	28.15	0.6181	179.37	6.99	2.2	31.43	2.3
18	174.41	27.14	0.9088	142.77	6.09	2.2	29.75	2.4
19	152.74	22.47	1.1432	114.45	5.12	2.3	24.69	2.6
20	135.10	22.77	1.2328	93.177	4.43	2.3	21.65	2.6
21	115.80	22.91	1.1749	77.143	4.14	2.4	24.36	2.7
22	96.10	19.75	1.1410	65.135	3.53	2.4	20.97	2.7
23	81.18	30.84	2.2585	54.701	3.2	2.5	31.50	3.4
24	68.79	28.71	2.2509	46.83	2.95	2.6	29.31	3.5
25	55.67	26.95	2.1414	40.307	2.64	2.6	27.46	3.4
26	43.62	25.36	2.2633	34.677	2.39	2.7	25.81	3.6
27	33.26	23.82	3.0656	30.094	2.17	2.7	24.21	4.1
28	28.08	20.73	4.4629	26.006	1.98	2.8	21.10	5.3
29	23.97	16.93	4.6898	22.49	1.79	2.9	17.30	5.5
30	20.86	12.35	4.7318	19.698	1.64	3.0	12.78	5.6

(a) [Q(3,6)] indicates that columns 3 and 6 are summed in quadrature.

(b) The uncertainty is composed of two main components: (1) the variability in the measurement of the secondary material; (2) the uncertainty in SRM 2806b. Most of the uncertainties are express at $k = 2$ level. The first four columns refer to the secondary material. Columns 5, 6 and 7 show the values and uncertainties associated with the SRM 2806b taken from the NIST Certificate of Analysis [10] and reported in the open literature [11] where column 6 is the $k = 1$ standard uncertainty in particle concentration and column 7 is the uncertainty in the particle diameter due to uncertainty in the concentration. Columns 8 and 9 are the $k = 2$ expanded total uncertainty found for the secondary calibration material in terms of particle concentration and in the particle diameter.

(c) [Q(4,7)] indicates that columns 4 and 7 are summed in quadrature.