National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2806b

Medium Test Dust (MTD) in Hydraulic Fluid

This Standard Reference Material (SRM) is intended for use in the calibration of instrumental response to medium test dust suspended in hydraulic fluid. A unit of SRM 2806b consists of two bottles containing polydisperse, irregularly shaped mineral dust suspended in approximately 400 mL of hydraulic fluid each.

Certified Values: Certification of this SRM is in terms of the projected area particle diameters of the collected dust particles from the hydraulic fluid. The diameters are made traceable to the NIST Line Scale Interferometer (LSI) through a NIST calibration of a Geller MRS-4XY pitch standard. The certified diameters are correlated with the numeric concentration of particles greater than each diameter, referred to as cumulative number particle size distribution. The mean cumulative particle concentrations versus certified diameter values from 1 µm to 30 µm are given in Table 1 and plotted in Figure 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy, all known or suspected sources of bias have been investigated or taken into account [1].

Information Values: Values given in Table 2 are provided for information only and are not certified. An information value is considered to be a value that may be of use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [1]. Information Values cannot be used to establish metrological traceability.

SRM 2806b can be used in conjunction with the International Organization for Standardization (ISO) method ISO 11171:2010, “Hydraulic Fluid Power - Calibration of Liquid Automatic Particle Counters” [2].

Expiration of Certification: The certification of **SRM 2806b** is valid, within the measurement uncertainty specified, until **31 December 2020**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see “Instructions for Handling, Storage, and Use”). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

The coordination of the technical measurements culminating in certification of SRM 2806b was led by R.A. Fletcher of the NIST Materials Measurement Science Division.

Design and scanning electron microscopy (SEM) imaging was performed by N.W.M. Ritchie. D.S. Bright developed the image processing software and provided the image analysis. R.A. Fletcher provided homogeneity testing, sample selection, preparation and data analysis. The aforementioned staff members are from the NIST Materials Measurement Science Division.

Statistical consultation including experimental design and uncertainty determination was provided by J.J. Filliben and W.F. Guthrie of the NIST Statistical Engineering Division.

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

John A. Small, Chief
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Gaithersburg, MD 20899
Certificate Issue Date: 12 June 2014

Robert L. Watters, Jr., Director
Office of Reference Materials

Source and Preparation[1]

The medium test dust, SAE 5-80 Micrometer Test Dust (Medium Test Dust) [3] lot number 4390C, used for the preparation of SRM 2806b was donated by Powder Technology Inc. (Burnsville, MN). Suspension of the dust in MIL-PRF-5606 hydraulic fluid was performed by Institut de la Filtration et des Techniques Separatives (IFTS) (Agen, France).

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

SRM 2806b should be mixed and sampled according to procedures described in ISO 11171:2010. Only if the sampled volume is decanted immediately after the prescribed mixing does the quantity of the SRM left in the original bottle remain certified (see "Sampling for Liquid Automatic Particle Counter" section). The SRM bottle should be kept upright and never heated above 80 °C and the bottle should be opened and sampled in a dust-free environment so as to avoid contamination.

Sampling for Liquid Automatic Particle Counter

Immediately before use, mix SRM 2806b in accordance with instructions in ISO 11171:2010. Briefly, the mixing process is as follows. Shake a closed bottle of SRM 2806b by hand or laboratory shaker for approximately 1 min. Disperse contents of bottle using an ultrasonic bath with minimum intensity of 5 W/cm^2 for 1 min. Mechanically shake for a minimum of 3 min on a commercial paint or laboratory shaker. Then ultrasonically treat briefly or evacuate to remove air bubbles from the suspension and introduce immediately into the liquid automatic particle counter. According to ISO 11171:2010 the minimum sample should be at least 10 mL and have 10 000 particles at the lowest threshold setting which should be obtained in each of at least 5 consecutive particle counts. Larger volumes are permissible. **CAUTION:** If the suspension is not used immediately after shaking and evacuation, large particles will settle to the bottom of the bottle and be omitted from the particle population measured.

NIST Certification Method[2]

The bottle-to-bottle homogeneity was determined using an HIAC ROYCO HR-LD 600 laser light extinction particle counter. The NIST measurements were compared to on-line and bottle-to-bottle measurements made by IFTS at the time of manufacturing. Material inhomogeneity was negligible with respect to the measurement uncertainty. The particle size and numbers measured for certifying this material were determined from digital images of dust particles extracted from aliquots of 8 bottles of the hydraulic fluid by filtration forming a set of 12 filters. These images were collected using a TESCAN MIRA3 Automated Field Emission Scanning Electron Microscope (SEM) operating in the backscatter and secondary electron modes. Particle area on the backscatter images was determined by image analysis that sums the pixels in a gray-level threshold (binary) domain that defines the particle [4]. The particle diameter is expressed as the projected area diameter of a stably oriented disc shaped particle (diameter of a circular particle with area equal to that of a particle in stable orientation).

Four SEM magnifications were used to obtain particle size data from particles ranging in size from 1 µm to 50 µm. A Geller MRS 4XY SEM Magnification Pitch Standard traceable to the NIST Line scale Interferometer [5] was used to determine the conversion of pixel dimension to metric values. More than 29 070 000 particles were sized from approximately 20 400 SEM image fields.

The measurand is the projected area particle diameter in micrometers. The certified values are metrological traceable to the SI unit of length through the NIST Line Scale Interferometer. The mean cumulative number particle concentration (particles/mL) in the hydraulic fluid is related to the projected area particle diameter values.

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[1] Certain commercial equipment, instruments or materials are identified in this certificate 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 instruments, materials, or processes identified are necessarily the best available for the purpose.

[2] The particle size was determined for the dust particles in their most mechanically stable orientation [6] and thus the particle area approaches, on average, the largest possible value. Literature values for the ratio of mean projected diameters for random and for stable oriented quartz particles (common to ISO Medium Test Dust) is approximately 0.8.

Table 1. Certified Values, Sources of Uncertainty, and Combined and Expanded Uncertainties for Projected-Area Particle Diameter in SRM2806b

Projected Area Particle Diameter[(a)] (µm)	Mean Cumulative Particle Concentration [(b)] (n = 12) (particles/mL)	Standard Uncertainty of Mean Cumulative Particle Concentration Due to Sampling Reproducibility[(c)] (n = 12) (particles/mL)	Standard Uncertainty in Projected Area Particle Diameter Due to Sampling Reproducibility[(c)] (n = 12) (µm)	Standard Uncertainty in Projected Area Particle Diameter Due to Image Digitization[(d)] (µm)	Combined Standard Uncertainty in Projected Area Particle Diameter (µm)	Coverage Factor, k[(e)]	Expanded Uncertainty, U, in Projected Area Particle Diameter (µm)
1	80 755	1 318.7	0.012 7	0.219 4	0.219 8	1.179 8	0.26
2	33 064	530.9	0.022 9	0.219 4	0.221 0	1.247 6	0.28
3	17 714	305.2	0.032 2	0.219 3	0.222 2	1.310 3	0.29
4	10 864	253.5	0.048 4	0.878 3	0.879 9	1.176 3	1.0
5	6 681.2	127.6	0.041 0	0.878 8	0.879 8	1.164 1	1.0
6	4 210.2	82.78	0.046 7	0.878 1	0.879 5	1.174 9	1.0
7	2 852.3	61.18	0.057 9	0.877 7	0.880 2	1.193 5	1.1
8	2 007.0	38.64	0.059 0	0.879 2	0.881 4	1.193 7	1.1
9	1 476.4	27.90	0.064 3	0.878 9	0.881 9	1.203 1	1.1
10	1 114.8	18.00	0.059 8	0.878 6	0.880 9	1.195 3	1.1
11	857.22	13.61	0.059 3	1.756 6	1.758 1	1.146 4	2.0
12	649.63	11.09	0.063 8	1.758 5	1.759 9	1.149 0	2.0
13	500.66	10.12	0.079 7	1.758 7	1.760 5	1.162 2	2.0
14	389.26	10.74	0.106 7	1.758 5	1.762 6	1.185 3	2.1
15	299.96	8.79	0.112 6	1.758 2	1.761 8	1.190 9	2.1
16	230.39	7.98	0.134 7	1.759 4	1.765 8	1.207 7	2.1
17	179.37	6.99	0.163 0	1.761 0	1.770 7	1.230 6	2.2
18	142.77	6.09	0.189 2	1.758 2	1.770 2	1.254 4	2.2
19	114.45	5.12	0.210 8	1.758 6	1.774 6	1.271 7	2.3
20	93.177	4.53	0.247 9	1.758 7	1.779 6	1.303 4	2.3
21	77.143	4.14	0.297 2	1.756 9	1.786 7	1.341 4	2.4
22	65.135	3.53	0.319 0	1.759 6	1.792 8	1.358 7	2.4
23	54.701	3.2	0.355 9	1.758 2	1.799 4	1.387 1	2.5
24	46.830	2.95	0.409 7	1.759 3	1.816 3	1.424 7	2.6
25	40.307	2.64	0.437 3	1.758 2	1.822 8	1.441 4	2.6
26	34.677	2.39	0.471 6	1.760 2	1.835 5	1.465 2	2.7
27	30.094	2.17	0.502 0	1.758 7	1.843 5	1.487 5	2.7
28	26.006	1.98	0.521 6	1.758 7	1.848 0	1.497 8	2.8
29	22.490	1.79	0.575 5	1.757 9	1.866 9	1.533 2	2.9
30	19.698	1.64	0.640 1	1.758 3	1.895 7	1.568 6	3.0

[(a)] Stable particle projected area diameter [6].
[(b)] Number of particles per milliliter of hydraulic fluid greater than the indicated diameter (number per milliliter).
[(c)] Type A uncertainties evaluated by statistical methods. The standard uncertainty in column 3 is the standard deviation in the cumulative particle concentration divided by the square root of 12 (n=12).
[(d)] Type B uncertainties evaluated by other means.
[(e)] *k* value determined by Monte Carlo calculation.
The expanded uncertainty for the projected area particle diameter corresponds to a 95 % confidence interval.

NIST Certified Uncertainties: The expanded uncertainties listed in Table 1 were calculated according to the JCGM GUM Supplement 1 [7]. These uncertainties correspond to the expanded uncertainties in particle projected area diameters and define a confidence interval of approximately 95 %. The combined and expanded uncertainties were derived using Monte Carlo simulation with inputs from the digitization and sampling reproducibility uncertainties.

The Type A and Type B standard uncertainty components arise from measurement uncertainties in the following sources: (1) particle sampling and counting, (2) fluid volume sampled, (3) SEM magnification and (4) digital image representation/processing. The uncertainty associated with volume is for the most part a Type A uncertainty and included in sampling results. The part that is not random is the uncertainty in the hydraulic fluid density which was determined to be approximately 8×10^{-5} g/mL and is too insignificant to impact the analysis. Similarly, the length and area uncertainty is associated with the uncertainty in the Geller Standard (0.002 µm and 0.0028 µm, respectively) and has little contribution to the total uncertainty in the measurements. The area uncertainty impacts the filter area which is only relevant to the sampling at high magnification since at low magnification the entire filter area was included.

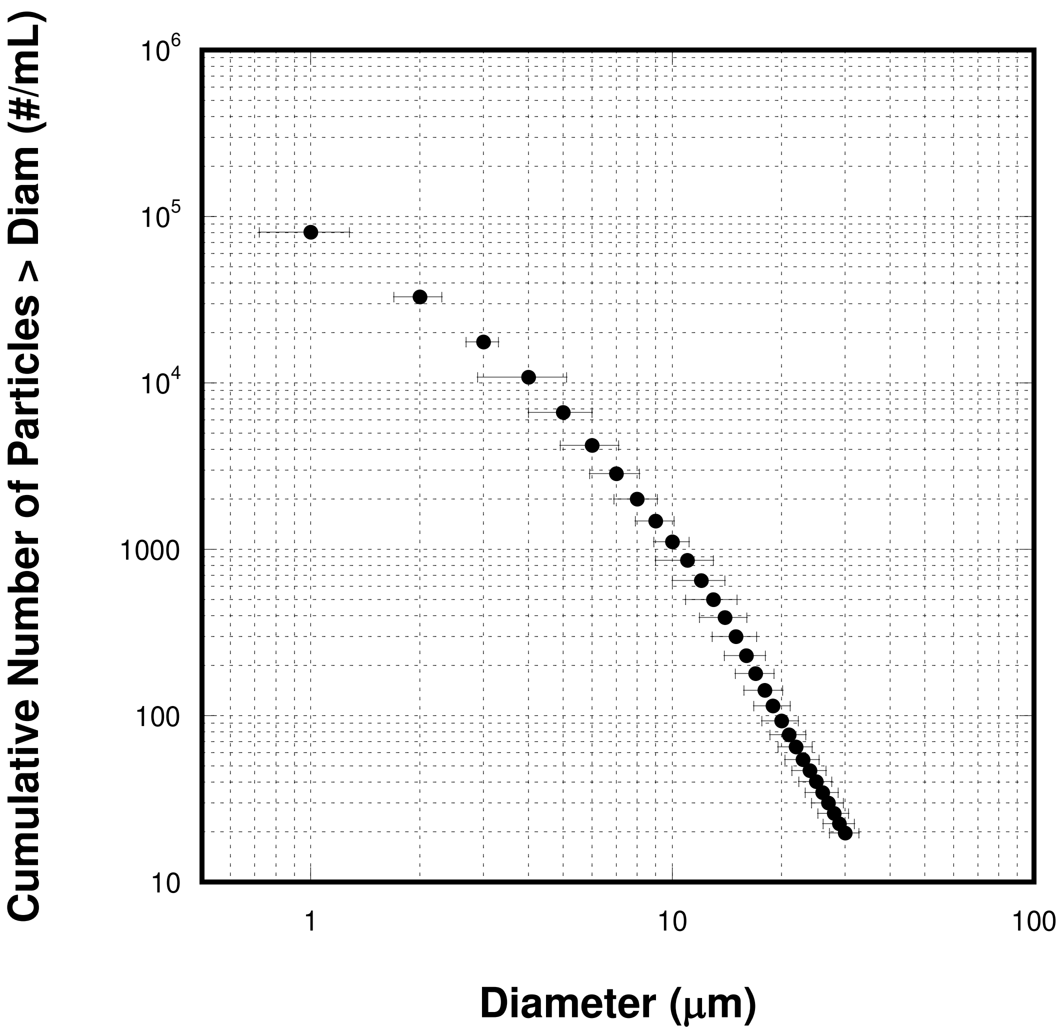

Figure 1. Plot of cumulative number of particles per milliliter of hydraulic fluid greater than the specified projected area particle diameter. The expanded uncertainties are shown in the plot.

Information Values: The values found in Table 2 are for information only. They were derived from a cubic spline fit to a plot of Mean Cumulative Particle Concentration values versus the Projected Area Particle Diameters. Table 2 is presented to provide additional estimates of cumulative particle concentrations at smaller projected diameter intervals and beyond 30 µm diameter range.

Table 2. Information Values for Number of Particles Per Milliliter of Hydraulic Fluid Greater Than the Projected Area Particle Diameter in SRM 2806b

Projected Area Particle Diameter (µm)	Mean Cumulative Particle Concentration (particles/mL)	Projected Area Particle Diameter (µm)	Mean Cumulative Particle Concentration (particles/mL)	Projected Area Particle Diameter (µm)	Mean Cumulative Particle Concentration (particles/mL)
1.0	80 755	11.5	745.66	34.0	11.775
1.1	75 186	12.0	649.63	34.5	11.026
1.2	69 665	12.5	569.03	35.0	10.301
1.3	64 241	13.0	500.66	35.5	9.655
1.4	58 963	13.5	441.57	36.0	9.073
1.5	53 878	14.0	389.25	36.5	8.524
1.6	49 037	14.5	342.17	37.0	8.007
1.7	44 486	15.0	299.95	37.5	7.526
1.8	40 276	15.5	262.76	38.0	7.087
1.9	36 453	16.0	230.39	38.5	6.689
2.0	33 066	16.5	202.77	39.0	6.304
2.1	30 149	17.0	179.37	39.5	5.909
2.2	27 667	17.5	159.72	40.0	5.540
2.3	25 574	18.0	142.77	40.5	5.228
2.4	23 821	18.5	127.73	41.0	4.942
2.5	22 358	19.0	114.44	41.5	4.643
2.6	21 138	19.5	102.98	42.0	4.342
2.7	20 112	20.0	93.176	42.5	4.060
2.8	19 232	20.5	84.789	43.0	3.811
2.9	18 449	21.0	77.145	43.5	3.598
3.0	17 714	21.5	70.765	44.0	3.394
3.1	16 974	22.0	65.134	44.5	3.180
3.2	16 233	22.5	59.695	45.0	2.973
3.3	15 498	23.0	54.704	45.5	2.793
3.4	14 772	23.5	50.483	46.0	2.626
3.5	14 059	24.0	46.831	46.5	2.457
3.6	13 366	24.5	43.463	47.0	2.309
3.7	12 695	25.0	40.307	47.5	2.199
3.8	12 051	25.5	37.362	48.0	2.094
3.9	11 440	26.0	34.678	48.5	1.962
4.0	10 865	26.5	32.287	49.0	1.826
4.5	8 049.6	27.0	30.094	49.5	1.717
5.0	6 681.2	27.5	27.999	50.0	1.626
5.5	5 345.9	28.0	26.006		
6.0	4 210.9	28.5	24.154		
6.5	3 414.3	29.0	22.489		
7.0	2 852.3	29.5	21.034		
7.5	2 389.3	30.0	19.697		
8.0	2 007.2	30.5	18.376		
8.5	1 710.5	31.0	17.137		
9.0	1 476.4	31.5	16.057		
9.5	1 279.8	32.0	15.075		
10.0	1 114.9	32.5	14.139		
10.5	978.31	33.0	13.273		
11.0	857.22	33.5	12.506		

REFERENCES

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Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.