National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2806a

Medium Test Dust (MTD) in Hydraulic Fluid

This Standard Reference Material (SRM) is intended for use in the calibration of instrument response to medium test dust suspended in hydraulic fluid. A unit of SRM 2806a consists of a two-bottle set of a polydisperse, irregularly shaped mineral dust suspended in approximately 400 mL of hydraulic fluid at a nominal concentration of 2.8 mg/L.

Certified Values: Certification of this SRM is in terms of the projected area diameters of the collected dust particles from the hydraulic fluid and the numeric concentration of particles greater than each diameter, referred to as cumulative number size distribution. Certified diameter values from 1 µm to 30 µm are given in Table 1a and plotted in Figure 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or taken into account [1].

Information Values: Values greater than 30 µm given in Table 1b are provided for information only and are not certified due to high measurement uncertainty. 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].

SRM 2806a can be used in conjunction with the International Organization for Standardization (ISO) method ISO/FDIS 11171, "Hydraulic Fluid Power - Calibration of Liquid Automatic Particle Counters" [2].

Expiration of Certification: The certification of **SRM 2806a** is valid, within the measurement uncertainty specified, until **31 December 2014**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified and exposed to excess heat (>80 °C).

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) will facilitate notification.

The coordination of the technical measurements leading to the certification of SRM 2806a was under the leadership of R.A. Fletcher of the NIST Materials Measurement Science Division.

Sample preparation, microscopy, and scanning electron microscopy (SEM) imaging were performed by J.R. Verkouteren, E.S. Windsor, J.A. Small, and E.B. Steel. D.S. Bright developed the image processing software and provided assistance. R.A. Fletcher provided homogeneity testing and SEM digital image processing data analysis. The aforementioned staff members are from the NIST Materials Measurement Science Division.

Statistical consultation was provided by W.S. Liggett formerly of the NIST Information Access and User Interfaces 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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Certificate Issue Date: 30 May 2013
Certificate Revision History on Last Page

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

SAMPLE PREPARATION[1]

The medium test dust, ISO 12103-A_3 (ISO Medium Test Dust) [3] lot number 4390C, used for the preparation of SRM 2806a was donated by Powder Technology Inc. (Burnsville, MN). Suspension of the dust in MIL-H 5606 hydraulic fluid was performed by Fluid Technology Inc. (Stillwater, OK).

INSTRUCTIONS FOR USE

SRM 2806a should be mixed and sampled according to procedures described in reference 2. 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 should not be heated above 80 °C and the bottle should be opened and sampled in a dust-free environment so as to avoid potential contamination.

Sampling for Liquid Automatic Particle Counter

Immediately before use, mix SRM 2806a in accordance with instructions in reference 2. Shake a closed bottle of SRM 2806a by hand for approximately 30 s. Ultrasonically disperse contents of bottle using a bath with minimum power of 5 W/cm^2. Mechanically shake for a minimum of 1 min on a commercial paint or laboratory shaker. Then ultrasonicate briefly or evacuate to remove air bubbles from the suspension and introduce immediately into the optical particle counter. **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 150 laser light extinction particle counter [4]. Material inhomogeneity was negligible with respect to the measurement uncertainty. The particle size [5] and numbers measured for certifying this material were determined from digital images of dust particles extracted from aliquots of four bottles of the oil by filtration. These images were collected using a SEM operating in the backscattered electron mode. Particle area was determined by "blobbing," an image processing method that sums the pixels in a gray-level thresholded (binary) domain that defines the particle [6]. The particle diameter is expressed as the projected area diameter of a stable oriented particle (diameter of a circular particle with an area equivalent to the area of a particle in stable orientation).

Five SEM magnifications were used to obtain particle size data from particles ranging in size from 1 µm to 50 µm. NIST SRM 484d SEM Magnification Standard was used to determine the conversion of pixel dimension to metric value. More than 775 000 particles were sized from approximately 4400 SEM image fields. Technical details of the measurement process are available in reference 4.

Round Robin Results: A round robin was conducted within the NFPA T2.9 Contamination Committee by three companies utilizing five instruments and five different sensors to compare counter and sensor response. SRM 2806a was measured using instruments that were calibrated in accordance with the existing ISO 4402:1991 Standard. The laboratories and their results are presented in Table 2.

[1]Certain commercial instruments, materials, or processes 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 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 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 1a. Certified Values, Sources of Uncertainty, and Combined and Expanded Uncertainties

Particle[(a)] Concentration (Particles/mL)	Projected[(b)] Area Diameter (µm)	u Sampling [(c)] (µm)	u Volume [(d)] (µm)	u Length [(c)] (µm)	u Digitization [(d)]	u Fractionation [(d)] (µm)	U_c Combined Standard Uncertainty (µm)	U Expanded Uncertainty (µm)
108 400	1	0.004	0.004	0.000 6	0.127	0.051	0.137	0.274
27 035	2	0.006	0.006	0.000 03	0.123	0.049	0.133	0.265
11 209	3	0.009	0.010	0.000 5	0.122	0.059	0.136	0.272
6 095	4	0.011	0.013	0.002	0.121	0.094	0.154	0.308
3 737	5	0.012	0.015	0.004	0.121	0.167	0.207	0.414
2 395	6	0.016	0.016	0.006	0.131	0.246	0.280	0.560
1 573	7	0.019	0.017	0.009	0.131	0.236	0.271	0.542
1 055	8	0.024	0.018	0.013	0.131	0.210	0.250	0.499
725.8	9	0.031	0.019	0.017	0.131	0.235	0.272	0.544
513.7	10	0.041	0.021	0.021	0.131	0.289	0.322	0.643
374.6	11	0.052	0.023	0.025	0.131	0.351	0.380	0.759
281.0	12	0.065	0.025	0.029	0.131	0.467	0.490	0.981
216.4	13	0.081	0.027	0.031	0.131	0.616	0.637	1.273
170.4	14	0.101	0.030	0.033	0.131	0.810	0.828	1.655
136.8	15	0.123	0.032	0.033	0.131	1.090	1.106	2.211
111.3	16	0.146	0.034	0.033	0.131	1.190	1.207	2.414
91.33	17	0.168	0.035	0.033	0.131	1.439	1.455	2.910
75.29	18	0.187	0.036	0.034	0.131	1.706	1.722	3.444
62.17	19	0.204	0.036	0.036	0.131	2.036	2.050	4.101
51.35	20	0.219	0.036	0.041	0.131	2.509	2.522	5.045
42.40	21	0.235	0.036	0.047	0.131	2.473	2.488	4.976
35.01	22	0.255	0.036	0.056	0.131	2.738	2.754	5.508
28.95	23	0.281	0.037	0.068	0.131	2.953	2.970	5.941
23.99	24	0.314	0.038	0.081	0.131	3.267	3.286	6.573
19.95	25	0.356	0.039	0.097	0.131	3.450	3.472	6.945
16.66	26	0.408	0.040	0.115	0.131	3.603	3.631	7.261
13.98	27	0.468	0.041	0.135	0.131	3.983	4.015	8.030
11.80	28	0.539	0.043	0.158	0.131	4.144	4.184	8.369
10.02	29	0.618	0.045	0.184	0.131	4.247	4.298	8.595
8.568	30	0.706	0.047	0.212	0.131	4.914	4.971	9.943

[(a)] Certified number of particles per milliliter of hydraulic fluid greater than the indicated diameter (number per milliliter) at approximately 25 °C.

[(b)] Stable particle projected area diameter [6], the standard uncertainty components (u), the combined standard uncertainties (U_c) and the expanded uncertainties (U) in the projected area diameter.

[(c)] Type A uncertainties evaluated by statistical methods.

[(d)] Type B uncertainties evaluated by other means.

NIST Certified Uncertainties: The expanded uncertainties listed in Table 1a were calculated according to the ISO/JCGM Guide [8]. These uncertainties correspond to the expanded uncertainties in particle projected area diameters, which are twice (coverage factor $k = 2$) the combined standard uncertainties. In cases where all uncertainties are Type A, then $k = 2$ level defines a confidence interval of approximately 95 %.

Each combined standard uncertainty is the square root of the sum-of-the-squares of the individual standard uncertainty derived or estimated from the measurement process. The Type A and Type B standard uncertainty components arise from measurement uncertainties in the following sources: (1) particle sampling and particle counting (Poisson counting), (2) fluid volume sampled, (3) SEM magnification, (4) digital image representation/processing, and (5) subsampling the bottle, which entails size fractionation due to fluid transfer.

Table 1b. Information Values for Number of Particles Per Milliliter of Hydraulic Fluid Greater Than the Indicated Diameter (number per milliliter) at Approximately 25 °C (Stable Particle Projected Area Diameter [6])

Particle Concentration (number/mL)	Diameter (µm)
7.380	31
6.403	32
5.594	33
4.921	34
4.356	35
3.880	36
3.476	37
3.131	38
2.834	39
2.577	40
2.354	41
2.159	42
1.988	43
1.836	44
1.702	45
1.581	46
1.474	47
1.377	48
1.289	49
1.209	50

Table 2. Participating Laboratories, Particle Counters and Optical Sensors Used for NFPA Round Robin
(Sensors Were Calibrated According to ISO 4402:1991)

			Particle Concentration[(b)] (number/mL Greater Than Indicated Size)						
Laboratory	Particle Counter	Sensor	1 µm (>4.2 µm)[(a)]	2 µm (>4.6 µm)	5 µm (>6.4 µm)	7 µm (>7.7 µm)	10 µm (>9.8 µm)	15 µm (>13.6 µm)	20 µm (>17.5 µm)
Nelson Industries, Inc.	Climet CI1000	RLV2 100H	5624	4429	2039	1215	576	191	77.9
Nelson Industries, Inc.	HIAC 9064	HR-LD 150	5351	4482	1977	1139	521	188	88.6
Fluid Technologies, Inc.	HIAC 8000	HR-LD 400	-------	4208	1898	1116	525	182	82.1
Pall Corp.	Met One 214	Met One 211W (0.5 µm to 25 µm)	5313	------	1993	------	541	182	82.5
Pall Corp.	Met One 214	Met One 211W (1 µm to 120 µm)	5350	------	1992	------	517	174	80.9

[(a)]Adjusted values in parentheses resulting from NIST calibration to SRM 2806a
[(b)]Values for particle concentration [number per milliliter (number/mL) greater than indicated size] are presented for information only.

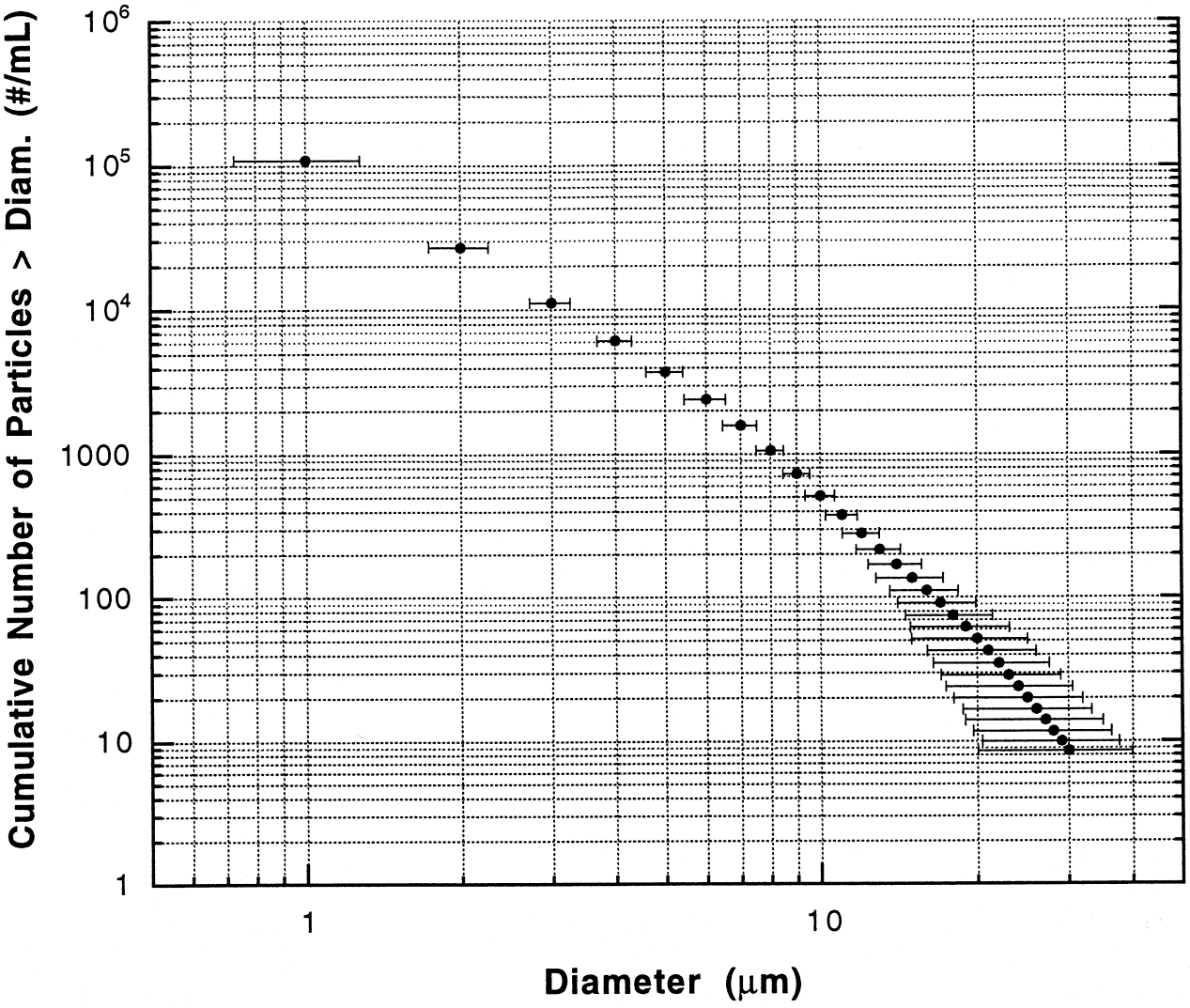

Figure 1. Plot of cumulative number of particles per milliliter of hydraulic oil greater than the specified projected area diameter. The uncertainty bars are the expanded uncertainties with $k = 2$.

REFERENCES

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[2] ISO 11171:2010; *Hydraulic Fluid Power–Calibration of Automatic Particle Counters for Liquids*; International Organization for Standardization: Geneva, Switzerland (2010).

[3] ISO 12103-1:1997; *Road Vehicles–Test Dust For Filter Evaluation–Part I*: *Arizona Test Dust*; International Organization for Standardization: Geneva, Switzerland (1997).

[4] Fletcher, R.A.; Verkouteren, J.R.; Windsor, E.S.; Bright, D.S.; Steel, E.B.; Small, J.A.; Liggett, W.S.; *SRM 2806a ISO Medium Test Dust in Hydraulic Oil*: *A Particle-Contamination Standard Reference Material for the Fluid Power Industry*; Fluid/Particle Separation Journal, Vol. 12, No. 2, pp. 80–95 (1999).

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[7] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed May 2013); 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://www.nist.gov/pml/pubs/index.cfm (accessed May 2013).

Certificate Revision History: 30 May 2013 (Extension of certification period; editorial changes); 01 November 2009 (Extension of certification period); 16 December 2008 (Extension of certification period); 29 January 2007 (Update of expiration date and editorial changes); 13 October 2004 (Original certificate date).

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.