National Institute of Standards & Technology

Certificate

Standard Reference Material® 2806d

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. The reference material should only be used with the document ISO 11171:2020 or a later version. A unit of SRM 2806d consists of three bottles containing polydisperse, irregularly shaped mineral dust suspended in approximately 400 mL of hydraulic fluid each. The certificate values are listed in see the Table 1 (below) or Table 2 (page 4).

Certified Values: The certified values of this SRM are related to a particle size metric of the medium test dust particles and were determined by consensus obtained through an international interlaboratory study (ILS). Certification of this SRM is in terms of the projected equivalent circular area particle diameters of the collected dust particles from the hydraulic fluid. The certified values (given below) are the diameters of circular particles with the same area found for the medium test dust particles and will be referred to in this certificate as particle diameter. The diameter values are made traceable to the NIST Line Scale Interferometer (LSI) through a NIST calibration of a Geller MRS-4XY pitch standard represented in SRM 2806b which in turn was used as the traceable calibrant for the ILS. The certified diameters are correlated with the cumulative particle concentration (particles/milliliter) of particles greater than each diameter, referred to as cumulative number particle size distribution. The measurand is the projected area particle diameter in micrometer(c) [µm(c)]. The median of the mean cumulative particle concentrations versus diameter values from 1.5 µm(c) to 30 µm(c) are given in Table 2 and plotted in Figure 1. Only certain projected diameters could be certified due to the low number of measured values provided by the ILS. The diameters that could be certified are illustrated in Table 1. A NIST-certified value is a value for which the NIST has the highest confidence in its accuracy and traceability; all known or suspected sources of uncertainty and bias have been investigated or considered [1]. It should be reemphasized that SRM 2806d should only be used in conjunction with the International Organization for Standardization (ISO) method ISO 11171:2020 or versions beyond 2020 of "Hydraulic Fluid Power — Calibration of Liquid Automatic Particle Counters" [2]. For calibration, refer to Table 2 that contains both certified and non-certified values.

Table 1. Certified Values for SRM 2806d

Projected Area Particle Diameter [µm(c)]	Median of Mean Cumulative Particle Concentration (part./mL)	Total Expanded Uncertainty of Median of Mean Cumulative Particle Concentration,(k = 2) (part./mL)	Combined Expanded Uncertainty, U, in Projected Area Particle Diameter in SRM 2806d, (k = 2) [µm(c)]
3	17 975.47	2 814.56	0.4
4	10 898.56	1 129.28	1.0
6	4 679.65	777.03	1.1
10	1 079.78	92.26	1.1
14	352.62	40.65	2.1
21	88.64	25.94	2.8
30	13.94	12.91	5.7

Expiration of Certification: The certification of **SRM 2806d** is valid, within the measurement uncertainty specified, until **31 December 2025**, 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.

R. David Holbrook, Chief
Materials Measurement Science Division

Gaithersburg, MD 20899
Certificate Issue Date: 16 March 2021

Steven J. Choquette, Director
Office of Reference Materials

Non-certified Values: There are 23 non-certified values shown in Table 2 that are provided for information only. They were derived from a mathematical fit calculation to individual sets of data. A non-certified 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]. Non-certified values cannot be used to establish metrological traceability.

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.

Coordination of the technical measurements culminating in certification of SRM 2806d was led by R.A. Fletcher and N.W.M. Ritchie of the NIST Materials Measurement Science Division. B. Verdegan, Cummins Filtration and 11171 Solutions (Stoughton, WI) was instrumental in all aspects of the organization, design and implementation of the interlaboratory study. NIST also acknowledges the generous contributions of thirteen laboratories located globally (listed below Table A1) that made the ILS possible by voluntarily providing analysis and reports of the candidate SRM 2806d.

Design and scanning electron microscopy (SEM) imaging was performed by N.W.M. Ritchie. D.S. Bright developed the image processing software that facilitated the image analysis. R.A. Fletcher provided material acquisition, homogeneity testing, consensus testing design, sample analysis in the interlaboratory study, image analysis and final data analysis. The aforementioned staff members are from the NIST Materials Measurement Science Division.

Statistical consultation including experimental design, consensus value and uncertainty determination was provided by J.J. Filliben of the NIST Statistical Engineering Division.

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

SRM 2806d should be mixed and sampled according to procedures described in ISO 11171:2020. Only if the sampled volume is decanted immediately (< 10 s) 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. The bottle should be opened and sampled in a dust-free environment so as to avoid contamination.

SOURCE, PREPARATION, AND ANALYSIS(1)

Source and Preparation: The medium test dust (MTD), SAE 5-80 Micrometer Test Dust (Medium Test Dust) [3] lot number 4390C, used for the preparation of SRM 2806d was donated by Powder Technology Inc. (Arden Hills, 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).

Sampling for Liquid Automatic Particle Counter: Immediately before use, mix SRM 2806d in accordance with instructions in ISO 11171:2020. Brief summary of the mixing process is as follows. Shake a closed bottle of SRM 2806d 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 briefly ultrasonically treat or evacuate to remove air bubbles from the suspension and introduce immediately into the liquid automatic particle counter. According to ISO 11171:2020 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 five 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. If air bubbles are not removed from the fluid, particle concentration determinations (number/mL) will be in error.

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(1)Certain commercial instruments, materials, or processes 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.

NIST Certification Method: SRM 2806d was certified as a consensus standard based on an ILS that was composed of 13 independent laboratories from five countries – China, France, Germany, United Kingdom and the United States. Instruments from three different manufactures and two different operating particle detection principles – light scattering and extinction, were used in the ILS. Some labs employed more than one instrument in the ILS and this provided 22 sets of data instead of only 13. Each laboratory had to qualify their instrument for the interlaboratory study.

The certified values in µm(c) are metrologically traceable to the SI unit of length through SRM 2806b which is traceable to the NIST Line Scale Interferometer [4]. The diameter is scaled to provide continuity back to the original SRM 2806 certified diameters. Based on studies in 2014, it was found that the diameters obtained from microscopy could be scaled by a factor of 0.898 to provide agreement with the original SRM 2806 certified diameters [5]. ISO TC131 SC6 WG1 has adopted the definition of micrometer(c) [µm(c)] [15]

$$1\ \mu\text{m(c)} = 0.898\ \mu\text{m(b)}$$

where µm(b) corresponds to the certified diameters reported in the certificate for NIST SRM 2806b [6].

The mean cumulative particle concentration (particles/mL > diameter) in the hydraulic fluid is related to the projected area particle diameter values presented in the certificate.

The bottle-to-bottle homogeneity was determined using an HIAC ROYCO HR-LD 600 laser extinction particle sensor. 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. There was no trend in the concentration with respect to production sequence.

The particle diameter and cumulative particle number concentration values measured for certifying SRM 2806d were traceable to SRM 2806b. SRM 2806b was NIST certified by digital analysis of scanning electron microscope images of medium test dust particles that were carefully extracted from aliquots of the SRM 2806b. 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 [7]. 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) [8]. A secondary calibration material traceable to SRM 2806b was made to specifications and according to ISO 11171 practices by the Aviation Industry (Xinxiang) Metrology and Test Science Technology Company Limited (CFPC, Xinxiang, China). Particle size distribution values were determined through a 4-laboratory study coordinated through the ISO committee T131 SC6 WG1 measuring the cumulative particle size distribution of the secondary material on automatic particle counters calibrated to SRM 2806b following procedures in ISO 11171:2016 [9]. The secondary calibrant test material traceable to SRM 2806b material was used as the only calibrant for the 13-lab ILS and thus the certified values for SRM 2806d are traceable through the ILS and ultimately to microscopic determinations associated to SRM 2806b.

Non-certified Values for SRM 2806d: Only seven diameters were populated with a sufficient number of measurements to allow certification, those values being (3, 4, 6, 10, 14, 21 and 30) µm(c). The remaining 23 diameters are provided in the certificate for information purposes only and are uncertified by the NIST. Median of the mean values were obtained from the consensus calculations similar to those shown in Figure A1. Each laboratory data set (Table A3) was fit to a mathematic model and interpolated values determined for each diameter based on the calculated results. The true measurement values, where there were ≥ 17 measurements (3, 4, 6, 10, 14, 21 and 30) µm(c), were preserved in the calculation, in the fitting process and in the reported values shown in this certificate. To reduce the confidence interval and the variance in the data, a two-step data transformation was undertaken. The uncertified values were derived from transforming the individual measurement values (n = 15 for each diameter) using Box–Cox transform methods [10–12] to normalize the variance at each diameter and thereby achieve a homoscedastic data set (equal variance). Then an additional Box–Cox transform was done to find the best functional form to linearize the data on an individual lab basis and then perform a linear fit for only that lab data. Data were fit to a linear model using the Bezier fitting code in the transform space and then values were transformed back into the measurement results space. The overall results are not identical but very similar, to those found from a Bezier constrained "cubic spline" fit to the same 22 data sets. All values in Table A4, since the raw measurement values were used in the model for each participant, represent the influence of each of participant's unique measurement processes and contribution to the consensus data set.

Table 2. Certified and uncertified reference values, sources of uncertainty, and Combined and Expanded Uncertainties for cumulative particle concentration and projected-area particle diameter in SRM 2806d[(a,b)].

1 Projected Area Particle Diameter [µm(c)]	2 Median of Mean Cumulative Particle Concentration for (part./mL)	3 Expanded Uncertainty of Median of Mean Cumulative Particle Concentration (part./mL)	4 Expanded Uncertainty in Projected Area Diameter [µm(c)]	5 Standard Uncertainty of Median of Mean Cumulative Particle Concentration for Secondary Calibration Material (part./mL)	6 Total Expanded Uncertainty in Projected Area Diameter for Secondary Calibration Material [µm(c)]	7 Total Expanded Uncertainty of Median of Mean Cumulative Particle Concentration [Q(3,5)][(c)] (part./mL)	8 Combined Expanded Uncertainty, *U*, in Projected Area Particle Diameter [Q(4,6)][(d)] [µm(c)]	9 Number of Measured Values
1.5	67 392.07	-[(f)]	-	-	-	-	-	1
2	41 165	2 485.3	0.08	1 076.24	0.3	2 708.32	0.3	4
3*	17 975.47	637.66	0.04	2 741.38	0.4	2 814.56	0.4	17
4*	10 898.56	255.4	0.05	1 100.02	1	1 129.28	1.0	22
5	6 986.32	117.97	0.04	884.52	1	892.35	1.0	4
6*	4 679.65	100.96	0.05	770.44	0.3	777.03	1.1	22
7	3 163.23	78.99	0.06	594.22	1.2	599.45	1.2	4
8	2 175.47	51.13	0.06	275.20	1.1	279.91	1.1	4
9	1 512.11	39.37	0.07	63.32	1.1	74.56	1.1	4
10*	1 079.78	30.34	0.08	87.13	1.1	92.26	1.1	22
11	786.62	24.73	0.10	81.63	2	85.29	2.0	4
12	586.06	23.88	0.14	61.06	2	65.56	2.0	4
13	449.93	21.31	0.18	42.30	2	47.36	2.0	4
14*	352.62	19.79	0.24	35.51	2.1	40.65	2.1	22
15	282.04	17.7	0.29	33.26	2.2	37.68	2.2	3
16	229.42	15.08	0.31	31.22	2.2	34.67	2.2	2
17	186.1	11.49	0.30	31.43	2.3	33.46	2.3	2
18	153.87	9.62	0.33	29.75	2.4	31.27	2.4	2
19	128.62	8.68	0.37	24.69	2.6	26.17	2.6	2
20	107.35	9.47	0.47	21.65	2.6	23.63	2.6	2
21*	88.64	8.92	0.56	24.36	2.7	25.94	2.8	21
22	75.56	6.98	0.52	20.97	2.7	22.10	2.8	2
23	61.94	7.21	0.58	31.50	3.4	32.31	3.4	2
24	50.86	6.58	0.65	29.31	3.5	30.04	3.6	2
25	41.61	5.06	0.58	27.46	3.4	27.92	3.4	2
26	33.44	3.89	0.50	25.81	3.6	26.10	3.6	2
27	26.08	3.1	0.48	24.21	4.1	24.41	4.1	2
28	20.41	2.56	0.53	21.0	5.3	21.25	5.3	2
29	16.39	2.12	0.66	17.30	5.5	17.43	5.5	2
30*	13.94	1.85	1.11	12.78	5.6	12.91	5.7	18

[(a)] The uncertainty in the distribution, column 3, was determined from a Bootstrap calculation for the certified values and by linear fit for the interpolated uncertified values (normal type), both at a $k = 2$. The uncertainty in cumulative particle concentration > diameter was given at the $k = 2$ level which is interpreted as approximately 95 % confidence level.

[(b)] Certified values and their associated uncertainties are denoted by gray and (*). These values were obtained from the consensus analysis that had 17 or more instruments reporting values.

[(c)] Q(3,5) indicates that columns 3 and 5 are summed in quadrature.

[(d)] Q(4,6) indicates that columns 4 and 6 are summed in quadrature.

[(e)] Column Definitions: **Column 1**: Stable particle projected area diameter [9]. **Column 2:** The consensus values (certified and non-certified) found from the ILS and reported as the median of the mean from all 22 laboratory measurements, cumulative particle concentration. **Column 3:** Uncertainty ($k = 2$) in the number of particles per milliliter greater than diameter evaluated by a Bootstrap and linear extrapolation calculation. **Column 4:** Uncertainty (k = 2) in the diameter due to the uncertainty in the particle concentration shown in Column 3. **Column 5**: Uncertainty in the diameter of secondary material traceable to SRM 2806b, $k = 2$ value determined by Monte Carlo calculation and propagation of uncertainty [Table A5, Col. 8]. **Column 6:** Uncertainty ($k = 2$) in the particle diameter for the secondary calibration material derived from Column 5, [Table A5, Col. 9]. **Column 7:** Total uncertainty ($k = 2$) associated with the particle concentration – sum in quadrature of measure uncertainties for ILS and for secondary material. **Column 8:** Total uncertainty ($k = 2$) in the projected area diameter at the level. **Column 9:** The number of independent measurements that went into arriving at the value.

[(f)] Uncertainty could not be determined where there is only one lab making one set of measurements.

NIST Uncertainties: The expanded uncertainties listed in Table 1, Table 2 and Table A5 were calculated according to the JCGM GUM Supplement 1 [13] and NIST SP 260-202 (2020) [14]. These uncertainties correspond to the expanded uncertainties in particle projected area diameters and particle concentrations; both define a confidence interval of approximately 95 %. The combined and expanded uncertainties were derived using Monte Carlo simulation with inputs from the uncertainty in the conversion factor 0.898 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) uncertainty found in the secondary calibration or test material which is traceable to the uncertainty in SRM 2806b. The uncertainty associated with sampling and counting is for the most part a Type A uncertainty and included in sampling results. The uncertainty in SRM 2806b (shown in certificate for SRM 2806b) is dominated by a Type B uncertainty related to the digital representation of the MTD particles in image analysis.

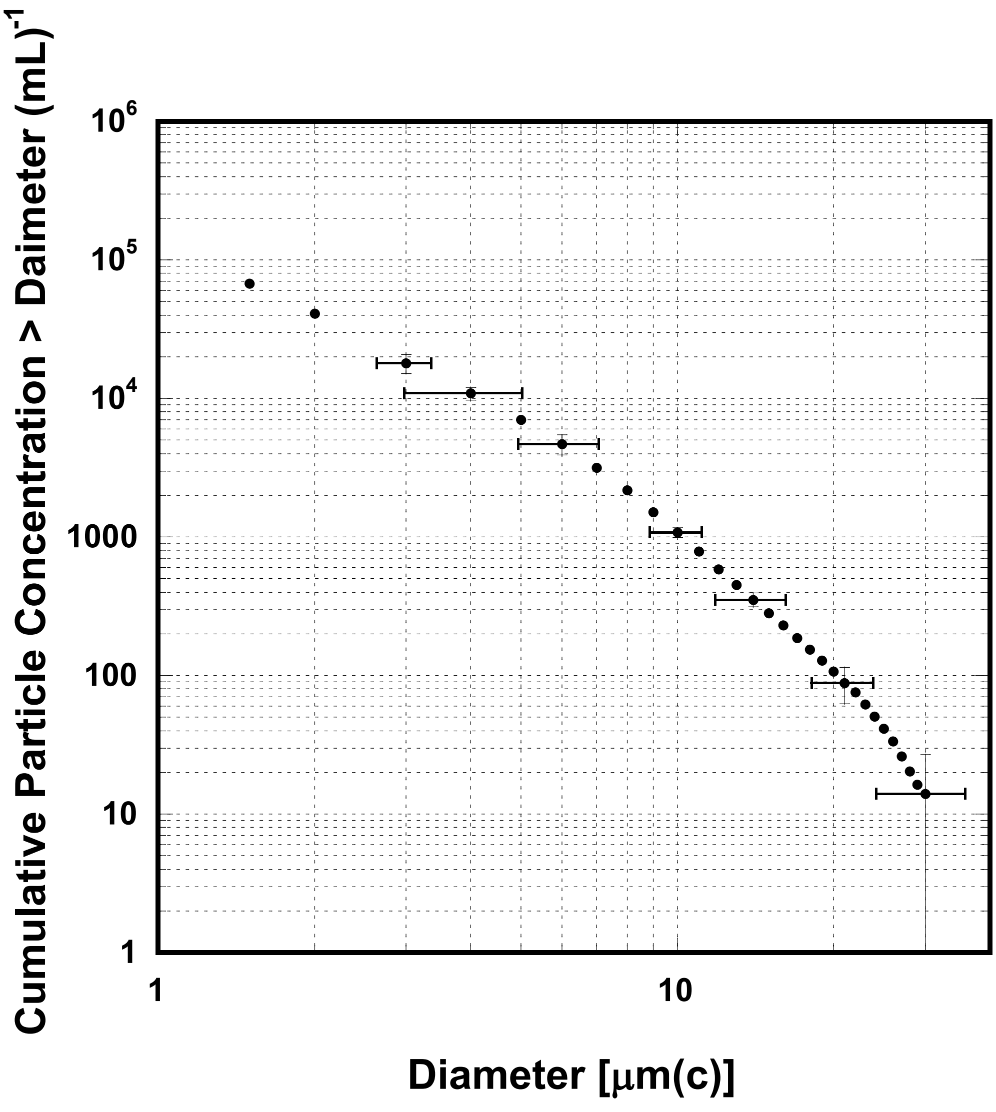

Figure 1. Plot of cumulative particle concentration greater than the specified projected area particle diameter. The expanded uncertainties for the certified diameters and for the cumulative particle concentration are shown in the plot.

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

APPENDIX A
Additional Information

An overview of the contributors/constituents is shown in Table A1. The identity of the participating laboratories was made anonymous by assignment of numerical identifiers (ID) 1 to 13. Particle Counter characteristics reported in Table A1 were derived through measurement in the ILS following procedures in ISO 11171:2020. Information presented in the columns is (1) measurement data set ID, (2) original lab ID, (3) IDs for the 13 labs in the study, (4) number of data points (diameters) reported, (5) manufacturer of automatic particle counter (APC) (there are three), (6) operating principle (either single particle scattering [S] or extinction [E]), (7) sensor noise level, (8) sensor coincidence limit in particles/mL, (9) sample volume reproducibility ($C_{V,vol}$), (10) sensor resolution determined with monodisperse polystyrene beads, (11) sample volume, (12) sample flow rate and (13) number of voltage thresholds possible or used in the measurement process. In column 2, original data set ID, if a lab analyzed one set of samples, they were assigned a single number and letter similar to 1a. A set of samples was three bottles of SRM 2806d. If a lab received two sets of samples, a lab received designation of, for example, 3a and 3b. Some labs used more than one particle counter, either a scattering or extinction sensor, which increased data sets up to 22. Part of the information in this table was reported in an ISO technical report [15] and was determined empirically in the 13 lab ILS

Table A1. Data Summary for the 13-Lab Interlaboratory Study

1. Meas. Dataset ID	2. Orig. Dataset ID	3. Lab ID	4. Number of Diameters Measured Reported	5. Instrument Manufact	6. Instru. Type	7. Instru. Noise Level (mV)	8. Coinciden Error (Part./mL)	9. $C_{V,vol}$ (%)	10. Instrument Resolution (%)	11. Sample Volume (mL)	12. Sample Flow Rate (mL/min)	13. Number Voltage Thresholds
1	1a	1	7	1	E	105	27 000	2.7	13.4	10	25	16
2	2a	2	7	2	E	10	19 000	0.4	5	10	50	12
3	3a	3	7	1	E	150	59 129	0.4	8.5	10	10	16
4	4a	3	7	1	S	80	12 713	0.2	6.8	10	10	16
5	3b	3	7	1	E	150	59 129	0.4	8.5	10	10	16
6	4b	3	7	1	S	80	12 713	0.2	6.8	10	10	16
7	5a	4	15	1	E	200	27 171	1.9	9.5	10	25	16
8	6a	5	7	1	E	120	89 813	0.8	9.6	10	10	18
9	6b	5	7	1	E	120	89 813	0.8	9.6	10	10	18
10	16a	5	15	1	S	40	14 003	0.8	10	10	10	18
11	7a	6	7	3	E	178	30 000	0.4	7.4	10	30	17
12	18a	6	5	3	E	181	30 000	0.4	7.4	10	30	17
13	8a	7	7	2	E	3	40 246	0.6	11.2	10	30	18
14	17a	7	7	2	E	8	15 789	0.6	7	10	20	18
15	9a	8	28	2	E	10	6 000	0.9	4.5	10	25	101
16	9b	8	28	2	E	10	6 000	0.9	4.5	10	25	101
17	10a	9	6	1	E	77	34 251	0.1	6.7	10	25	13
18	11a	10	7	1	E	190	19 418	0.2	9.6	10	25	16
19	12a	11	6	1	E	190	20 743	0.3	6	10	25	24
20	13a	11	7	1	S	150	12 245	0.6	3	25	10	24
21	14a	12	7	1	E	170	25 780	1	10.1	10	25	16
22	15a	13	6	1	E	155	19 617	0.9	5.8	10	25	16

Table A1 provides the information describing participants and the instruments used in the ILS. For example, measurement set 1 and 2 (1a, 2a) were collected by lab 1 and lab 2, each reporting data for seven particle diameters with lab 1 using an instrument from manufacturer 1 and lab 2 using manufacture's 2 instrument. Both used extinction to measure particle size, and each had a series of operating parameters described in the columns to the right that were determined by the participating labs during the ILS. Measurement sets 3 to 6 were obtained by laboratory 3, reporting seven diameters, using instrument 1 and extinction for two of the data sets and light scattering to measure the other two data sets.

Participating in the study were 13 laboratories from 12 companies and participants located globally that made the ILS possible by voluntarily providing analysis and reports of the candidate SRM 2806d: ARGO-HYTOS GMBH; Aviation Industry Metrology and Test Science Technology Co., Ltd.; Beckman Coulter; Cummins Filtration; Donaldson; Fluid Technologies, Inc.; Institut de la Filtration et des Techniques Separatives (IFTS); NIST; PAMAS; Parker Hannifin; Southwest Research Institute and Stanhope-Seta, Ltd.

Each participating laboratory was provided with at least three bottles of secondary calibration fluid and three bottles of candidate SRM 2806d material. Each bottle was selected to yield a three-bottle set with the properties of controlled, balanced coverage of the respective material, and provide a broad representative sample of the material in the study. After calibration of the automatic particle counter using only the secondary test material, each lab measured three bottles of candidate 2806d material in the prescribed manner. Results from the measurements were collected by the National Fluid Power Association and collated by B. Verdegan, Cummins Filtration. The data was transferred to NIST and analyzed using DATAPLOT software developed by J. Filliben, NIST, [11] to determine the consensus values. An example of the methods used to determine the consensus values is shown in Figure A1 for the cumulative particle concentration results found at 10 µm(c). The left side in Figure A1 shows the results for the 22 sets of measurements of the cumulative particle concentration. The $k = 1$ pooled variation (standard deviation) within each lab is shown to be about 3.4 %. The lab-to-lab agreement is less than ideal (between lab standard deviation is approximately 7 %). The labs are not in statistical agreement as shown by the significant ANOVA FCDF result, indicating that the labs are not drawing from populations with the same mean value. The non-agreement of the labs was shown to be the case not just for this 10 µm(c) diameter, but for all particle diameters. This is thought to be due to the combined effect of particle counter calibration and measurement variation between participants; extensive homogeneity testing indicated that the material is bottle-to-bottle homogeneous at approximately 1.5 % level at 10 µm(c).

The right side of the plot presents the consensus value calculated by 14 different statistically accepted methods. The definition of each method is found in Table A2. It is relevant that all methods of determining the consensus value are in good agreement as shown from the plot and the tabulated results found in the far-right margin. The uncertainty U ($k = 2$) for each consensus value is tabulated. For this ILS the median of the mean (MedM) has been selected as the statistic-of-choice due to its outlier-resistant properties, for example, at 10 µm(c); (1079.9 ± 30.2) particles/mL because it best represents the data even in the presence of wide variation. The uncertainties for each method are shown at the far right of Figure A1 and are found using a Bootstrap methodology.

Cumulative Particle Concentrations: Values for cumulative particles concentrations were tabulated in Table A3 from consensus calculations like those shown in Figure A1. All of the mean values ($n = 15$) are shown for each laboratory. Only one lab provided a value at 1.5 µm(c), four labs for 2 µm(c), 17 labs for 3 µm(c), 22 labs for (4, 6, 10, 14) µm(c), 21 labs for 21 µm(c) and 18 labs for 30 µm(c). The actual measurement data is fairly sparse as shown in Table A3. To expand the information beyond measurement values and thus augment the size distributions, each set of laboratory data (each column) was modeled by 10 different methods, several of which used the Bezier constrained cubic fit interpolation program code actually used in the ILS, to interpolate missing values. The interpolation results are shown in Table A4. Since each data set is fit independently, characteristics or properties of the measurement process for each lab is preserved. No extrapolation was done. Median of means were determined from modeled data like that shown in Table A4. For the diameters where there were ≥ 17 measurements, the true measurement values from the ILS were preserved.

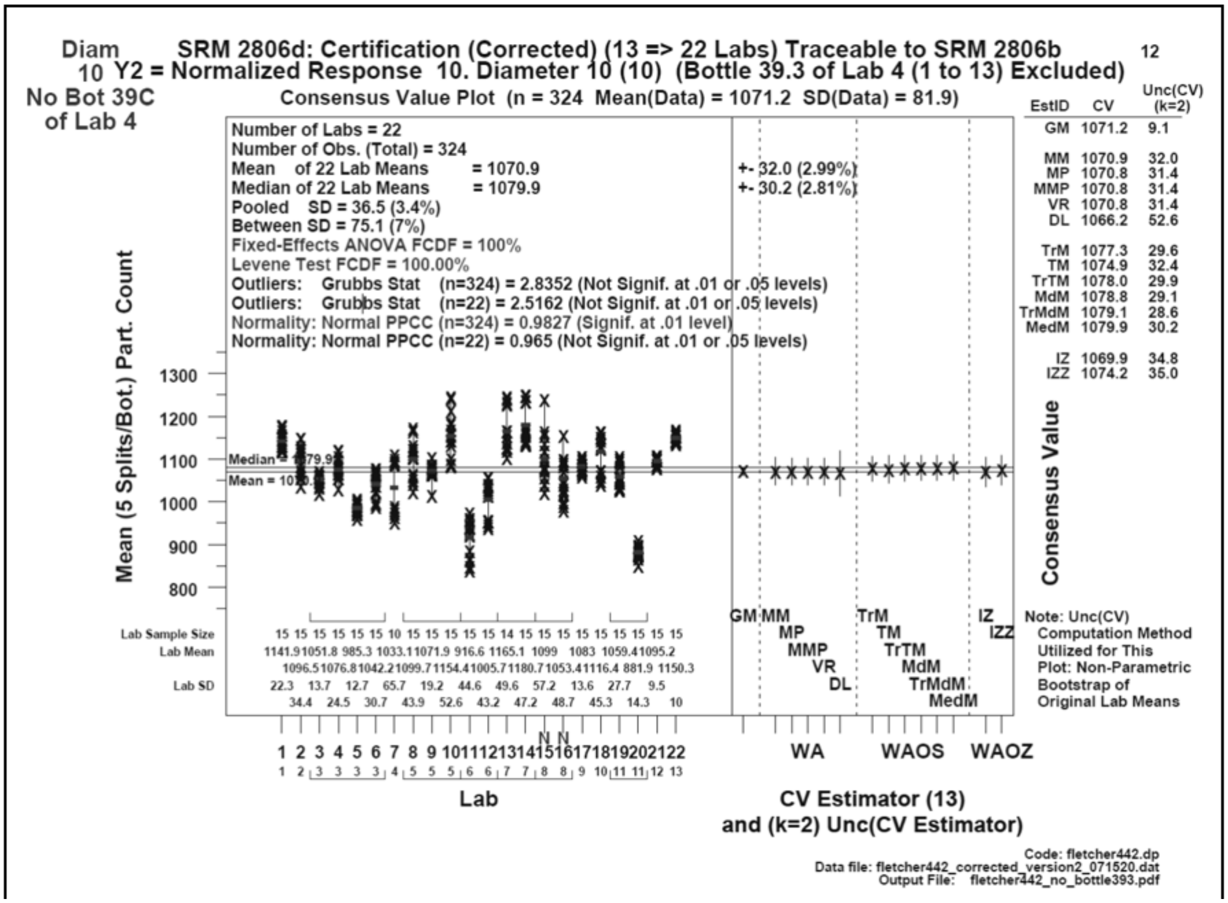

Figure A1. Example of the ILS data for 10 µm(c). The abscissa presents the data sets 1 to 22 (labs 3, 5, 6, 7, 8 and 11 each provided two or more measurement results giving 22 data sets in total). The ordinate gives the cumulative particle concentrations. Each analysis set consisted of three bottles with five repetitions for each bottle (15 total measurements). The spread in the data is apparent with the mean value for each lab shown as a bar. NIST was lab 15 and 16 and analyzed two sets or six bottles. The uncertainty shown in the far right was derived from a Bootstrap method to model the variation inherent in the data.

Table A2. Definitions of the 14 consensus-value estimators shown in Figure A1

GM	Grand Mean
MM	Mean of Means (Compute weights, CV, and Unc(CV))
MP	Mandel-Paule (Compute weights, CV, and Unc(CV))
MMP	Modified Mandel-Paule (Compute weights, CV, and Unc(CV))
VR	Vangel-Rukhin (Compute weights, CV, and Unc(CV))
DL	DerSimonian-Laird (Compute weights, CV, and Unc(CV))
TrM	(1,1) Trimmed Mean of Means (Compute weights, CV, and Unc(CV))
TM	Triangular Mean of All Means (Compute weights, CV, and Unc(CV))
TrTM	(1,1) Trimmed Mean of Means (Compute weights, CV, and Unc(CV))
MdM	(1,1) Trimmed Triangular Mean of Means (Compute weights, CV, and Unc(CV))
TrMdM	Midmean of Means (Compute weights, CV, and Unc(CV))
MedM	Median of the Means (Compute weights, CV, and Unc(CV))
IZ	Inverted mean
IZZ	Inverted Mean **2

Table A3. Mean values for cumulative particle concentration > diameter (particles/mL) compiled from the available measurements from the ILS, 13 laboratories and 22 separate determinations. Each value represents the mean of 15 measurements at a particular particle diameter [µm(c)] shown on the far-left column. Within NIST link: NIST Report of Analysis # 643-02-20, Table 8 (page 23) and statistician report of analysis exhibit 3.30 (page 80).

Orig Lab ID:	1a	2a	3a	4a	3b	4b	5a	6a	6b	16a	7a	18a	8a	17a	9a(NIST)	9b(NIST)	10a	11a	12a	13a	14a	15a		
New Dataset ID (22):	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**	**19**	**20**	**21**	**22**		
Physical Lab (13):	**1**	**2**	**3**	**3**	**3**	**3**	**4**	**5**	**5**	**5**	**6**	**6**	**7**	**7**	**8**	**8**	**9**	**10**	**11**	**11**	**12**	**13**		
Device within Lab:	1	1	1	2	1	2	1	1	1	2	1	2	1	2	1	1	1	1	1	2	1	1		
Diameter																							**#Labs**	**CV**
1.5										67392.07													1	67392.07
2				42237.40		41248.87				41081.13										37154.33			4	41165.00
3	19122.33	19793.60	17424.47	17975.60	17546.40	17325.20	17500.30	18333.60	17676.00	19016.80			18097.86	18537.80	16667.34	16357.67		18210.40		16382.33	19738.60		17	17975.60
4	11207.73	11337.13	10666.20	10491.67	10613.73	10103.33	10649.60	11220.53	10849.07	11412.53	10590.00	10869.40	10928.64	11099.53	10322.96	10048.69	11423.13	10957.07	11074.93	9556.93	10969.53	11229.40	22	10899.02
5							6910.60			7598.00					6825.59	6631.87							4	6868.09
6	4839.13	4723.73	4592.67	4982.93	4469.07	4842.13	4502.40	4727.40	45[illegible]1.00	4867.93	4247.40	4502.20	4689.71	4727.53	4515.33	4386.35	4935.60	4677.00	4650.00	4065.07	4682.73	4778.80	22	4679.87
7							3070.90			3318.47					3098.51	2987.37							4	3084.70
8							2112.44			2373.43					2173.73	2094.01							4	2143.09
9							1457.05			1666.98					1526.31	1471.32							4	1498.81
10	1141.89	1096.53	1051.83	1076.76	985.33	1042.16	1033.07	1099.67	1071.93	1154.41	916.62	1005.70	1165.07	1180.73	1099.03	1053.42	1083.01	1116.41	1059.42	881.89	1095.15	1150.27	22	1079.88
11							725.53			871.05					805.79	761.28							4	783.53
12							520.19			643.04					602.01	564.10							4	583.05
13							397.38			479.41					472.47	444.44							4	458.46
14	353.42	325.97	357.40	318.80	310.67	319.84	319.75	354.53	338.20	377.65	299.35	318.91	392.93	402.80	380.26	352.55	356.59	375.33	348.65	265.02	382.41	362.06	22	352.98
15							248.58								304.69	284.37							3	284.37
16															244.59	232.20							2	238.40
17															191.16	184.15							2	187.65
18															155.04	149.12							2	152.08
19															134.25	129.78							2	132.02
20															117.67	114.17							2	115.92
21	86.35	84.13	96.51	69.80	80.19	80.32	78.04	94.67	84.33	100.42	67.78	79.09	103.91	107.80	98.42	95.83		105.52	87.93	55.52	109.62	89.51	21	87.93
22															79.50	78.38							2	78.94
23															68.37	66.97							2	67.67
24															56.69	55.70							2	56.19
25															46.21	43.48							2	44.84
26															37.25	33.93							2	35.59
27															27.57	24.19							2	25.88
28															22.64	19.63							2	21.14
29															19.47	16.72							2	18.10
30	11.87	12.40	11.47		10.59		10.82	14.07	12.87		13.89	12.38	20.09	17.07	16.49	14.56	27.44	18.01	14.37		17.03	12.40	18	13.98
# Diameters:	7	7	7	7	7	7	15	7	7	15	6	6	7	7	28	28	5	7	6	7	7	6		

Table A4. Mean values after interpolation for cumulative particle concentration > diameter (particles/mL) from measurement process and the model-calculated values. The original measurement values at the high participation diameters (3, 4, 6, 10, 14, 21 and 30) µm(c) are preserved in this data matrix and highlighted in the table.

Orig Lab ID	1a	2a	3a	4a	3b	4b	5a	6a	6b	16a	7a	18a	8a	17a	9a(NIST)	9b(NIST)	10a	11a	12a	13a	14a	15a		
Data Set ID	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**	**19**	**20**	**21**	**22**		
Physical Lab	**1**	**2**	**3**	**3**	**3**	**3**	**4**	**5**	**5**	**5**	**6**	**6**	**7**	**7**	**8**	**8**	**9**	**10**	**11**	**11**	**12**	**13**		
Device within Lab	1	1	1	2	1	2	1	1	1	1	1	2	1	2	1	1	1	1	1	2	1	1		**Model 10**
Diameter																							**# cells**	**Values**
1.5										67389													1	67 392.07
2				42237		41249				41079										37153			4	41 165
3	19121	19792	17424	17975	17546	17325	17477	18333	17675	19016			18097	18538	16663	16354		18209		16382	19739		17	17 975.47
4	11207	11336	10666	10492	10614	10103	10635	11220	10848	11412	10589	10869	10928	11099	10320	10046	11423	10956	11074	9556.8	10970	11229	22	10 898.56
5	7205.9	7141.8	6866.2	7100.8	6758.4	6864	6901.2	7149.7	6934.5	7597.3	6541.6	6839.1	7002.1	7076.9	6822.9	6630	7374	7011.9	7007.5	6110	6970.5	7150.3	22	6 986.32
6	4839	4723.2	4592.6	4982.8	4469	4841.9	4496.7	4727.1	4590.6	4867.3	4246.7	4501.4	4689.4	4727.1	4513.1	4384.9	4935.5	4676.6	4649.8	4065	4682.7	4778.8	22	4 679.65
7	3282.6	3182.3	3092.2	3373.4	2981	3277.3	3067	3185.6	3097	3317.9	2787.9	2996.1	3207.6	3232.6	3096.6	2986.3	3283.2	3169.9	3110.6	2706.1	3156.6	3248.2	22	3 163.23
8	2257.6	2183.6	2097.1	2254.1	2005.8	2180.7	2109.5	2178.6	2122.6	2372.8	1861.8	2024.7	2231.5	2252.2	2172.3	2093	2197.8	2180.5	2109.1	1816.5	2152.7	2245.1	22	2 175.47
9	1586.3	1530.5	1460.7	1532.7	1384.2	1480.4	1454.9	1526.4	1489.1	1666.3	1282.8	1405	1591.3	1609.3	1525.1	1470.3	1514.3	1537.8	1470.7	1247.3	1509.9	1587.8	22	1 512.11
10	1141.8	1096.2	1051.8	1076.6	985.29	1041.9	1031.4	1099.2	1071.8	1153.8	915.9	1005.1	1164.6	1180.3	1098.1	1052.7	1082.9	1115.9	1059.2	881.82	1095.1	1150.2	22	1 079.78
11	831.79	790.02	778.15	773.53	716.46	752.41	724.3	805.5	783.23	870.54	673.08	733.77	865.65	879.78	805.04	760.66	794.76	825.61	780.4	636.53	815.81	842.39	22	786.62
12	611.54	572.8	587.62	563.1	529.47	551.96	519.22	598.53	579.08	642.52	504.7	543.8	651.4	664.35	601.43	563.49	593.88	620.23	584.5	466.05	620.16	622.18	22	586.06
13	458.6	424.73	453.35	418.32	400.19	414.29	396.61	454.44	436.85	478.9	385.34	411.42	499.59	511.22	471.91	443.6	454.23	476.04	446.52	347.62	481.69	468.47	22	449.93
14	353.3	325.77	357.38	318.73	310.58	319.63	319.15	354.4	338.16	377.03	299.02	318.79	392.05	402.25	379.81	351.95	356.56	374.77	348.47	264.89	382.39	362.05	22	352.62
15	280.24	259.09	288.56	248.72	248.07	253.14	247.97	284.44	269.06	305.31	235.41	253.45	314.92	324.13	304.16	283.84	285.98	303.18	278.18	205.88	311.04	287.81	22	282.04
16	227.03	211.24	237.64	197.31	202.55	204.26	200.16	233.23	218.45	249.93	187.57	205.56	257.35	265.97	244.07	231.8	232.72	250.37	226.1	162.31	258.12	233.51	22	229.42
17	186.31	174.85	197.97	158.41	167.58	167.07	164.74	193.75	179.62	206.27	150.93	168.79	212.74	220.72	190.71	183.78	191.67	209.4	185.89	129.35	216.73	191.85	22	186.1
18	153.91	145.82	165.81	128.26	139.57	137.98	136.94	162.02	148.7	171.24	122.43	139.55	177.13	184.27	154.57	148.82	159.5	176.28	153.84	103.93	183.03	158.64	22	153.87
19	127.34	121.81	138.98	104.42	116.42	114.71	114.09	135.71	123.37	142.74	99.98	115.65	148.05	154.18	133.9	129.55	133.89	148.68	127.69	83.99	154.75	131.44	22	128.62
20	105.1	101.46	116.15	85.28	96.88	95.75	94.66	113.4	102.21	119.29	82.09	95.77	123.88	128.87	117.33	114.02	113.24	125.19	106	68.15	130.55	108.71	22	107.35
21	86.26	83.94	96.47	69.71	80.15	80.09	77.81	94.23	84.3	99.81	67.71	79.02	103.56	107.32	98.1	95.66	96.41	104.89	87.8	55.43	109.57	89.48	22	88.64
22	70.36	68.98	79.59		65.92		63.34	77.84	69.21		56.1	64.94	86.52	89.03	79.26	78.15	82.55	87.46	72.54		91.43	73.28	22	75.56
23	57.16	56.44	65.26		53.98		51.24	64.06	56.67		46.68	53.23	72.34	73.66	68.19	66.81	71.04	72.7	59.81		75.91	59.79	18	61.94
24	46.25	45.99	53.13		43.96		41.26	52.49	46.27		38.99	43.52	60.47	60.72	56.5	55.53	61.4	60.22	49.22		62.7	48.59	18	50.86
25	37.27	37.32	42.85		35.58		33.11	42.81	37.67		32.66	35.5	50.48	49.84	45.99	43.22	53.28	49.7	40.41		51.48	39.31	18	41.61
26	29.9	30.17	34.17		28.57		26.49	34.73	30.58		27.43	28.89	42.06	40.69	36.85	33.79	46.39	40.86	33.09		41.99	31.63	18	33.44
27	23.88	24.29	26.85		22.73		21.16	28	24.74		23.08	23.45	34.94	33	27.1	23.83	40.52	33.43	27.01		33.99	25.3	18	26.08
28	18.98	19.46	20.72		17.88		16.89	22.42	19.95		19.45	18.98	28.9	26.55	22.11	19.32	35.49	27.21	21.96		27.26	20.09	18	20.41
29	15.01	15.53	15.61		13.87		13.48	17.81	16.02		16.42	15.33	23.78	21.15	18.78	16.35	31.16	22.02	17.78		21.63	15.83	18	16.39
30	11.8	12.33	11.38		10.57		10.76	14.02	12.82		13.86	12.34	19.44	16.65	15.8	14.2	27.41	17.69	14.32		16.94	12.35	18	13.94

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 A5 summarizes the properties of the secondary material and provides the total uncertainty associated with the secondary material. This uncertainty is propagated throughout the results.

Table A5. 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)][b]	9 U 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) The uncertainty is composed of two main components: (1) the variability in the measurement of the secondary material; (2) the uncertainty in SRM 2806b. All 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 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.

(b) Q(3,6) indicates that columns 3 and 6 are summed in quadrature.

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