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Hydraulic fluid power — Background, impact and use of ISO 11171:2020 on particle count and filter test data

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 131, *Fluid power systems*, Subcommittee SC 6, *Contamination control*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The 2020 revision of ISO 11171 was initiated due to depletion of supplies of the National Institute of Standards and Technology (NIST) Standard Reference Material® (SRM) 2806b, which is required for primary calibration of liquid automatic particle counters (APC) using ISO 11171:2016. The 2016 edition of ISO 11171 also provides an option for reporting particle size in units of either µm(c) or µm(b), which has resulted in confusion among users of particle count data. µm(b) sizes are about 10 % larger than the corresponding µm(c) sizes. Thus, µm(b) concentrations can be as much as 8 times (3 ISO Codes) lower, and µm(b) filter Beta Ratios can be an order of magnitude lower than the same numerical value reported in µm(c). This is problematic when attempting to conform with fluid cleanliness and filter performance specifications.

ISO 11171:2020 addresses these issues by specifying the historically consistent, traceable µm(c) as the sole acceptable means of reporting particle size. Unlike the 2016 edition, ISO 11171:2020 is not dependent upon a specific batch of SRM 2806, as NIST henceforth certifies the material as a consensus standard to minimize the potential for shifts in particle size with future batches. Additional refinements to ISO 11171 facilitate calibration at smaller and larger particle sizes.

Hydraulic fluid power — Background, impact and use of ISO 11171:2020 on particle count and filter test data

1 Scope

This document provides the background for ISO 11171:2020 and the use of µm(c) as the sole means of reporting particle size for APC particle count data. It also summarizes results of the international inter-laboratory study (ILS) of its reproducibility using SRM 2806d candidate material and suspensions of Reference Material (RM) 8632a. The ILS results provided the basis for certification of SRM 2806d used for primary calibration of APC. Their implications with respect to particle counting and filter testing are discussed in this document.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3534-1, *Statistics — Vocabulary and symbols — Part 1: General statistical terms and terms used in probability*

ISO 3534-2, *Statistics — Vocabulary and symbols — Part 2: Applied statistics*

ISO 3534-3, *Statistics — Vocabulary and symbols — Part 3: Design of experiments*

ISO 4406, *Hydraulic fluid power — Fluids — Method for coding the level of contamination by solid particles*

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 11171, *Hydraulic fluid power — Calibration of automatic particle counters for liquids*

ISO 16889, *Hydraulic fluid power — Filters — Multi-pass method for evaluating filtration performance of a filter element*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3534-1, ISO 3534-2, ISO 3534-3, ISO 4406, ISO 5725-1, ISO 11171 and ISO 16889 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Undesirable consequences of ISO 11171:2016

ISO 11171:2016 specified the use of NIST SRM 2806b for primary APC sizing calibration. Prior to this, SRM 2806 and SRM 2806a, which have the same certified particle size distribution, were used for primary calibration. Supplies of SRM 2806 and SRM 2806a were exhausted by 2010. The replacement batch, SRM 2806b, was released to the market in 2014. SRM 2806 and SRM 2806b were certified by scanning electron microscopy (SEM), but SRM 2806b was produced by a different supplier and advanced methods of metrology were used. An important difference between the batches is that the images of particles for SRM 2806 were manually processed, while SRM 2806b used automated image analysis. Particle sizes obtained by APC calibrated with SRM 2806b were found to be about 10 % larger than sizes obtained using SRM 2806 or SRM 2806a calibrations that yielded the same particle number concentration. This 10 % difference in size is significant and prompted the revision of ISO 11171.

In response to the particle size shift, ISO 11171:2016 introduced an alternative method for reporting particle size, µm(b). Prior to 2016, APC calibrated with ISO 11171 reported particle size in units of µm(c). With ISO 11171:2016, users had the option to report size in units of µm(c) or µm(b), whereby µm(c) sizes were obtained by multiplying µm(b) sizes by a factor of 0,898 and are numerically equivalent to the previous µm(c). Users wanting to report sizes directly related to the NIST SRM 2806b SEM results could report data as µm(b) sizes. Users attempting to meet existing cleanliness levels or filter performance specifications or desiring historical consistency in the data could report as µm(c) sizes.

The alternative methods of reporting particle size resulted in confusion. There was an unfounded belief that µm(b) sizes were more accurate, but this is not supported by statistical analysis. There is no evidence that µm(b) sizes are closer to the true particle sizes than the µm(c) sizes obtained using SRM 2806 or SRM 2806a. Regardless, some chose to report µm(b) sizes, while others used µm(c). This is problematic when vendors and customers, or analytical laboratories and end-users report in different units of size. For example, if fluid cleanliness is specified in terms of an ISO 4406 Code at 4, 6 µm(c) and 14 µm(c), but the APC reports data in 4 µm(b), 6 µm(b) and 14 µm(b), how can it be decided if fluid is clean enough? According to ISO 11171:2016, 4 µm(b), 6 µm(b) and 14 µm(b) sizes correspond to 3,6 µm(c), 5,4 µm(c), and 12,6 µm(c), but there is no mathematical relationship to directly relate particle concentrations. The problem is compounded if the conversion between µm(b) and µm(c) sizes was calculated incorrectly or if the measurement units for particle size were mis-labelled. While ISO 11171:2016 provided a convenient alternative means for converting particle size, it resulted in confusion that needed to be addressed.

5 Rationale for ISO 11171:2020

Like the previous editions, ISO 11171:2020 retains traceability to the internationally accepted definition of a metre. Unlike the 2016 edition, ISO 11171:2020 allows only a single method of reporting particle size in units of µm(c). Reporting size in units µm(b) is no longer an option. The ISO 11171:2020 µm(c) is equivalent to the historical µm(c) obtained using SRM 2806. It no longer specifies a specific batch of SRM 2806 for primary calibration and does not need to be revised with each new batch of SRM 2806x. ISO 11171:2020 includes other changes, including a standardized method for creating APC calibration curves, the use of dilution to facilitate calibration at small sizes, and a standardized method for calibrating at sizes larger than 30 µm(c).

ISO 11171:2020 uses samples with an NIST certified particle size distribution, NIST SRM 2806x, for primary calibration. Certification provides a measure of the true value of the particle concentration at different sizes, but there is uncertainty associated with any measurement. For SRM 2806 and SRM 2806b, sources of uncertainty include the number of bottles analysed, bottle to bottle differences, sub-sampling from a bottle, fractionation on the membrane used for SEM, particle orientation on the membrane, digitization, pixilation, and measurement of length. The certified concentrations for each batch of SRM are likely to be near the median particle concentration, but a different measure of the median is likely to be obtained each time a new batch is certified. Thus, there is likely to be a particle size shift with each new batch of SRM 2806 certified in this manner. The challenge is to reduce the size shift to insignificance.

Beginning with SRM 2806d, NIST will certify SRM 2806x as a consensus standard to reduce the potential for a shift in particle size. Previously, SRM 2806 and SRM 2806b were certified by SEM analysis of the calibration fluid, but there are many sources of uncertainty resulting in the apparent particle size shift between batches. In contrast, a consensus standard, like SRM 2806d, is developed in co-operation with all parties with an interest in participating in the development or use of the standard. In this case, NIST and ISO TC 131/SC 6 agreed to use the traceable SRM 2806 certification to define µm(c) and APC (rather than SEM) data to obtain the number concentration of particles as a function of particle size. To avoid commercially significant shifts in particle size between batches, the certified particle size distribution for SRM 2806x batches are based upon particle count data from ILS conducted using APC calibrated according to ISO 11171:2020 in µm(c) using the most recent previous batch of SRM 2806x. In the case of SRM 2806d, SRM 2806b was used to calibrate all 4 APCs used to certify the ILS secondaries as per ISO 11171:2020 and to maintain traceability. Samples of SRM 2806b had been put aside by NIST at the request of the Project Leaders specifically for this purpose. The traceable secondaries were used to calibrate ILS APCs. In this manner, the ILS data represents the industry consensus definition of µm(c) and ensures that particle size does not deviate significantly with each new batch of SRM 2806x.

In addition to changes in the manner of reporting particle size and certification of primary calibration suspension, ISO 11171:2020 also specifies how calibration curves are determined. Previously, there was discretion in the way calibration curves were determined, e.g. how channels were selected, whether to use latex or test dust to calibrate for sizes greater than 30 µm(c), what mathematical function is used to create a calibration curve. Collectively, these legitimate discretionary choices increased uncertainty in particle count data and, in some cases, introduced artefacts into the data. This is addressed in ISO 11171:2020, which requires that data from at least 12 different threshold settings spaced logarithmically over the entire particle size range of interest be used to create a calibration curve. The lowest of these threshold settings is 1,5 times the threshold noise level of the instrument, corresponding to the smallest size that can be counted. The calibration curve itself is created using the constrained cubic spline method. In this manner, the uncertainty between laboratories is reduced.

Counting particles smaller than 4 µm(c) has been problematic, as the concentrations of particles in calibration suspensions are typically above coincidence error limits. In contrast to prior versions, ISO 11171:2020 permits dilution of calibration samples using a defined method derived from ISO 11500. To minimize contamination, verification of dilution fluid and glassware cleanliness is required. As a result, some laboratories participating in the ILS were able to calibrate to particle sizes as small at 1,5 µm(c).

In some applications, such as gear and transmission fluids, interest is primarily in particles larger than 30 µm(c). Previous versions of ISO 11171 allowed either latex or test dust calibration suspensions to be used at these sizes, but they can yield different results. Furthermore, test dust calibration samples generally contain insufficient numbers of large particles for a valid calibration. Also, primary calibration samples are not certified above 30 µm(c) and therefore not traceable. To address this, ISO 11171:2020 specifies that primary calibration for particle sizes larger than 30 µm(c) be done with monodispersed latex particles and provides guidance for selection of their sizes. APC calibrated in this manner can be used to produce secondary calibration suspensions certified at sizes larger than 30 µm(c). These changes are intended to reduce uncertainty and to facilitate calibration at particle sizes larger than 30 µm(c).

6 Inter-laboratory study experimental design

An inter-laboratory study (ILS) of ISO 11171:2020 was conducted to:

— Measure the intra-company repeatability and inter-company reproducibility of particle count data obtained using APC calibrated to the standard using SRM 2806d candidate material and suspensions of RM 8632a;

— Generate particle count data to be used by NIST to certify consensus standard SRM 2806d in size units of µm(c);

— Determine the extent, if any, of the shift in particle size resulting from the use of SRM 2806d; and

— Generate particle count data for NIST RM 8632a that will provide a basis to update ISO 11171:2020, Table A.1.

The ILS experimental design consisted of:

1) Production of traceable secondary calibration samples for use by ILS participants.

2) Selection and qualification of participating laboratories and APC.

3) ISO 11171:2020 calibration of APC.

4) Analysis of samples of SRM 2806d candidate suspensions and RM 8632a test dust, and

5) Analysis of data as per ISO 5725-2.

The ILS results were provided to NIST for use in certifying consensus standard SRM 2806d.

The traceable secondary calibration samples for use in the ILS were prepared by Aviation Industry (Xinxiang) Metrology and Test Science Technology Company Limited, commonly known as CFPC. These were used to calibrate the APC used by ILS participants. They were produced to the same specifications as the SRM 2806d candidate material according to ISO 11171:2020, Annex F. Four laboratories were selected to analyse the secondary calibration samples. Two of the laboratories, Pamas and CFPC, utilized Pamas light scattering APC. All data for particle sizes of 1,5 µm(c) and 2 µm(c) were generated by Pamas light scattering APC. The other two laboratories, NIST and Beckman, utilized Beckman light extinction APC. Each laboratory performed a full primary ISO 11171:2020 calibration but using SRM 2806b for particle sizes up to 30 µm(c) for light extinction sensors or to the largest size that the instrument was capable of counting for light scattering sensors. Each was sent five bottles of secondary calibration fluid for analysis. The data from all four laboratories was analysed by NIST and used to generate the composite certified size distribution for the secondary calibration samples. Thus, the certified size distribution is not biased with respect to a single laboratory, APC operating principle or manufacturer. ILS participants were required to demonstrate that their APC met all ISO 11171 performance specifications and asked to provide their most recent ISO 11171:2016 calibration curve. Eighteen APC participated in the ILS, including laboratories from five countries (USA, Germany, United Kingdom, China, France), two different operating principles (light extinction, light scattering), and three APC manufacturers (Pamas, Beckman, Stanhope-Seta).

The characteristics of these 18 individual APC are summarized in Table 1.

Table 1 — APC operating and performance characteristics

APC identifier	APC manufacturer	Type	Noise level mV	Coincidence error limit Particles/mL	CV, vol %	Resolution µm(c)	Sample volume mL	Working flow rate mL/min	Number of thresholds
1	X	extinction	105	27 000	2,7 %	13,4 %	10	25	16
2	Y	extinction	10	19 000	0,4 %	5,0 %	10	50	12
3	X	extinction	150	59 129	0,4 %	8,5 %	10	10	16
4	X	scattering	80	12 713	0,2 %	6,8 %	10	10	16
5	X	extinction	200	27 171	1,9 %	9,5 %	10	25	16
6	X	extinction	120	89 813	0,8 %	9,6 %	10	10	18
7	Z	extinction	178	30 000	0,4 %	7,4 %	10	30	17
8	Y	extinction	3	40 246	0,6 %	11,2 %	10	30	18
9	Y	extinction	10	6 000	0,9 %	4,5 %	25	25	101
10	X	extinction	77	34 251	0,1 %	6,7 %	10	25	13
11	X	extinction	190	19 418	0,2 %	9,6 %	10	25	16
12	X	extinction	190	20 743	0,3 %	6,0 %	25	25	24
13	X	scattering	150	12 245	0,6 %	3,0 %	10	10	24
14	X	extinction	170	25 780	1,0 %	10,1 %	10	25	16
15	X	extinction	155	19 617	0,9 %	5,8 %	10	25	16
16	X	scattering	40	14 003	0,8 %	10,0 %	10	10	16
17	Y	extinction	8	15 789	0,6 %	7,0 %	10	20	18
18	Z	extinction	181	30 000	0,4 %	7,4 %	10	30	17

ILS participants calibrated their APC as per ISO 11171:2020 using the CFPC secondary calibration samples previously described. Participants were asked to calibrate to the smallest size that their APC was capable of counting and, if possible, to calibrate up to a particle size of 50 µm(c) or the largest size that their APC was capable of counting. Each participant received 3 samples of SRM 2806d candidate material and 1 bottle of RM 8632a to analyse. The candidate material samples were analysed as described in ISO 11171:2020 at as many of the following particle sizes as possible using their ISO 11171:2020 calibration: 1,5 µm(c), 2 µm(c), 3 µm(c), 4 µm(c), 6 µm(c), 10 µm(c), 14 µm(c), 21 µm(c), 30 µm(c), 38 µm(c) and 50 µm(c). Participants were also asked to prepare three 1,0 mg/L samples of RM 8632a test dust and analyse these at as many of the integral particle sizes as possible between 2 µm(c) and 15 µm(c), inclusive. The results were provided to the Project Leaders and analysed as per ISO 5725-2. The data and statistical analysis were also provided to NIST for use in certifying the consensus standard SRM 2806d. Further details regarding the ILS and the certification of SRM 2806d are described in Reference [6].

7 Results of ILS

Tables 2 and 3 summarize the ILS data for the SRM 2806d candidate material and RM 8632a samples, respectively. Figures 1 and 2 show the mean particle concentration data from each ILS participant for the samples in graphical form. As per ISO 5725-2, Mandel h and k, Grubbs test, and Cochran test were used to detect outlier data. Outlier data are noted in Tables 2 and 3 and were excluded from the statistical analysis.

The following are suspected outliers, but did not meet the criteria for exclusion from the data analysis:

— 6 µm(c), 10 µm(c) and 14 µm(c) data of participant 13 for the SRM 2806d candidate samples;

— 38 µm(c) and 50 µm(c) data of participant 7 for the SRM 2806d candidate samples;

— 3 µm(c), 7 µm(c) and 8 µm(c) data for the second RM 8632a sample analysed by participant 9;

— 6 µm(c) through 15 µm(c) data of participant 1 for their RM 8632a sample; and

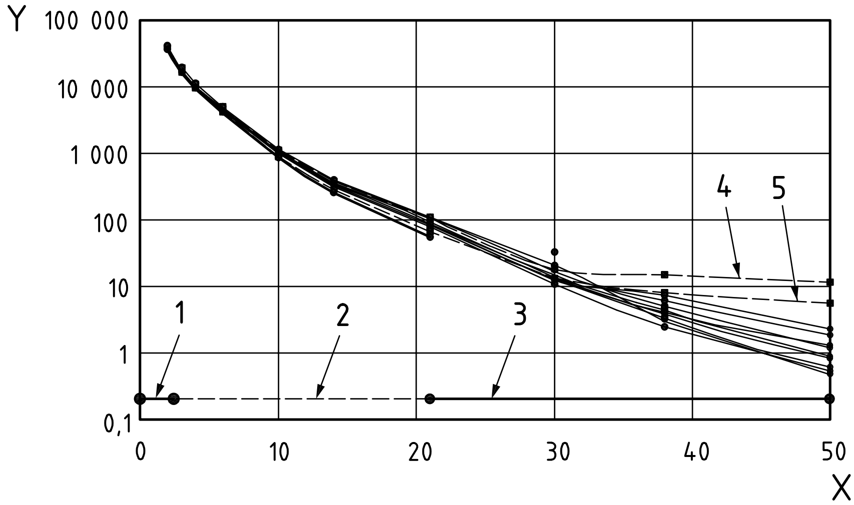
— 3 µm(c) data of participants 16 and 17 for their RM 8632a sample.

Referring to Figure 1, the SRM 2806d candidate material data at particle sizes larger than 30 µm(c) for participants 7 and 11 are suspected outliers. Statistical grounds exist to exclude participant 11, but not participant 7 at these sizes. In both cases, the particle concentration data appeared to approach a horizontal asymptote at larger sizes and the corresponding APC calibration curve also demonstrated asymptotic behaviour. In some cases, the latex calibration samples were reported to be cloudy in appearance, suggesting that the water drops from the latex did not completely disperse in the dilution fluid, thus causing the APC to count water droplets. This results in the asymptotic behaviour at these sizes. The root cause appears to be incomplete dissolution of the Aerosol OT in the dilution fluid. Dissolution requires heat and time and can be difficult to verify visually. Participant 6 initially encountered a similar issue, but resolved it by using a higher dilution ratio, i.e. a lower latex concentration in the calibration samples. Another approach is to increase the heating and stirring time used to dissolve Aerosol OT in the dilution fluid, thus ensuring complete dissolution. Guidance on how to avoid this issue was added to ISO 11171:2020 based upon these results.

Referring to Figure 2, RM 8632a data for participants 1 and 5 were suspected to be outliers. While participant 1 consistently exhibited the lowest RM 8632a data, it did not meet the outlier criteria and was not excluded. Participant 5 data was excluded as an outlier. These data were approximately twice the mean value of all other laboratories at each size, suggesting that a math or weighing error occurred, but this could not be confirmed.

Table 2 — Statistical summary of ILS data for SRM 2806d candidate material samples

Particle size, µm(c)	Number of labs	Outliers	Mean, particles/mL	Standard deviation, particles/mL			CV, %		
				Repeatability	Between labs	Reproducibility	Repeatability	Between labs	Reproducibility
1,5	2		67 419,93	614,10	0,00	614,10	0,91 %	0,00 %	0,91 %
2	3		40 430,44	505,51	2 402,69	2 455,30	1,25 %	5,94 %	6,07 %
3	12	5	18 014,44	276,26	1 098,51	1 132,71	1,53 %	6,10 %	6,29 %
4	16	5,13	10 862,58	164,49	401,09	433,50	1,51 %	3,69 %	3,99 %
6	17	5	4 642,08	86,18	222,73	238,82	1,86 %	4,80 %	5,14 %
10	18		1 071,04	37,40	74,13	83,03	3,49 %	6,92 %	7,75 %
14	18		346,64	20,62	32,02	38,08	5,95 %	9,24 %	10,99 %
21	17		88,20	8,61	13,53	16,04	9,77 %	15,34 %	18,19 %
30	13	10,11	14,05	3,30	2,17	3,95	23,50 %	15,43 %	28,12 %
38	12	11	4,59	1,46	1,61	2,17	31,83 %	35,04 %	47,34 %
50	11	11	1,45	0,68	1,36	1,52	47,02 %	93,26 %	104,45 %



Key

Y particle/mL> indicated size

X particle size, µm(c)

1 sizes covered only by light scattering sensors

2 sizes covered by light scattering and extinction sensors

3 sizes cover only by light extinction sensors

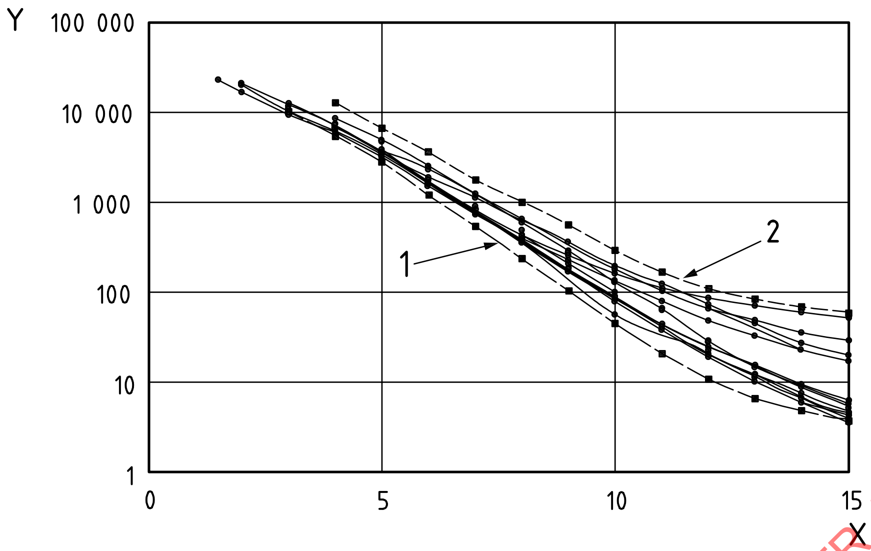
4 participant 11

5 participant 7

Figure 1 — Mean particle concentration versus particle size results for SRM 2806d candidate material samples

Table 3 — Summary of RM 8632a ILS data

particle size, µm(c)	Number of labs	Outliers	Mean, particles/mL	Standard deviation, particles/mL			CV, %		
				Repeatability	Between labs	Reproducibility	Repeatability	Between labs	Reproducibility
2	3		19 377,69	127,58	2 217,84	2 221,51	0,66 %	11,45 %	11,46 %
3	11		10 527,80	547,04	775,94	949,39	5,20 %	7,37 %	9,02 %
4	16	5,9b	6 561,31	68,52	763,56	766,62	1,04 %	11,64 %	11,68 %
5	16	5,9b	3 597,02	55,14	531,32	534,18	1,53 %	14,77 %	14,85 %
6	16	5,9b	1 761,37	44,51	356,31	359,08	2,53 %	20,23 %	20,39 %
7	16	5	844,71	51,68	182,14	189,33	6,12 %	21,56 %	22,41 %
8	16	5	425,40	37,85	105,11	111,71	8,90 %	24,71 %	26,26 %
9	14	5	218,68	28,14	63,83	69,76	12,87 %	29,19 %	31,90 %
10	17	5	113,21	18,62	41,06	45,09	16,45 %	36,27 %	39,83 %
11	13	5	62,74	14,29	31,33	34,43	22,78 %	49,94 %	54,89 %
12	15	5	34,01	10,39	22,11	24,43	30,53 %	65,01 %	71,82 %
13	13	5	23,27	8,91	19,02	21,00	38,28 %	81,74 %	90,26 %
14	16	5	15,53	6,62	14,48	15,92	42,62 %	93,26 %	102,54 %
15	15	5	11,22	5,21	13,09	14,09	46,47 %	116,68 %	125,60 %



Key

Y particle/mL> indicated size

X particle size, µm(c)

1 participant 1

2 participant 5

Figure 2 — Mean particle concentration versus particle size results for 1,0 mg/L RM 8632a samples

ISO 11171:2020 exhibits reduced uncertainty compared to ISO 11171:2016. This is shown in Table 4 using data from Unknown A for the 2015 ILS and the SRM 2806d candidates. The 2 types of samples are similar, but not identical. Test dusts used to prepare the samples had similar particle size distributions, but they were prepared by different laboratories, using different preparation methods and different concentrations. The differences are expected to have minimal impact on the between-laboratories coefficient of variation (CV) and reproducibility but can impact the within laboratory repeatability. Referring to Table 4, the 2019 ILS exhibits lower (better) between laboratories CV and reproducibility at all particle sizes, especially at sizes 30 µm(c) and smaller. The between laboratories CV with ISO 11171:2020 ranges between 5 and 26 % lower than the 2016 version of the standard. The reproducibility with ISO 11171:2020 ranges between 3 and 16 % lower than the 2016 version. The within laboratory repeatability ranges from 1,0 % better to 17 % worse, depending upon the particle size. This is believed to reflect differences between the samples used. Overall, the ILS results suggest ISO 11171:2020 to exhibit significantly lower uncertainty than the 2016 version.

Table 4 — Comparison of CV results from 2015 and 2019 ILS

Particle size µm(c)	2015 ILS CV, %			2019 ILS CV, %		
	Repeatability	Between labs	Reproducibility	Repeatability	Between labs	Reproducibility
4	2,56 %	10,3 %	10,7 %	1,51 %	3,7 %	4,0 %
6	2,27 %	13,9 %	14,1 %	1,86 %	4,8 %	5,1 %
10	2,51 %	23,7 %	23,8 %	3,49 %	6,9 %	7,8 %
14	2,82 %	25,5 %	25,6 %	5,95 %	9,2 %	11,0 %
21	4,65 %	20,5 %	21,0 %	9,77 %	15,3 %	18,2 %
30	8,98 %	42,0 %	42,9 %	23,5 %	15,4 %	28,1 %
38	14,6 %	53,7 %	55,6 %	31,8 %	35,0 %	47,3 %

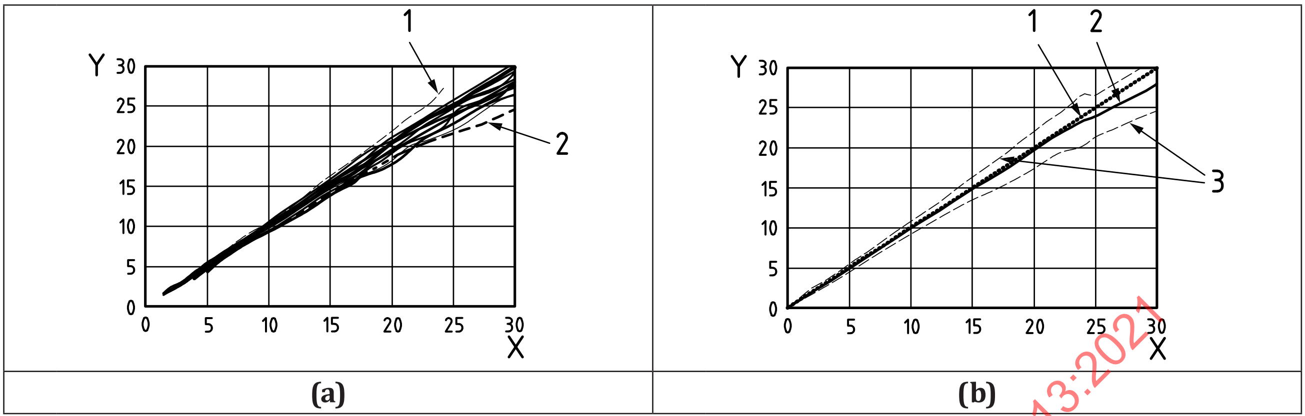
8 Impact of ISO 11171:2020 on particle count data

To assess whether SRM 2806d produced a shift in particle size (as was observed upon the release of SRM 2806b), 2019 ILS calibration curves and the most recent ISO 11171:2016 calibration curves from 16 participating APCs were obtained. Using the constrained cubic spline method and the most recent 2016 calibration curves, threshold voltage settings corresponding to integral µm(c) sizes for each of these APC were determined. The sizes that correspond to these threshold settings were then determined using the 2019 ILS calibration curve for the same APC. In this manner, 2016 and corresponding 2019 sizes for each threshold voltage setting for each APC were determined. The data were examined for outliers and the remaining data used to determine the average particle size shift for each size over the range from 1,5 µm(c) to 30 µm(c). It is noteworthy that the 2019 data are the basis for the consensus certification of SRM 2806d and are expected to correspond closely to the final certified sizes.

This analysis suggests that the size shift will be approximately 5 times less than was observed when SRM 2806b was introduced. A comparison of 2016 and 2019 sizes (see Table 5) shows that the average particle size shift over the particle size range from 2 µm(c) to 30 µm(c), inclusive, is -1,7 %. When SRM 2806b was introduced, a size shift of 10,2 % was observed. The ILS analysis includes APC from 3 different APC manufacturers, and both light extinction and light scattering sensors. For particle sizes 2 µm(c) and smaller, data was only obtained from light scattering sensors. For particle sizes between 3 and 21 µm(c), inclusive, data was obtained from both light extinction and light scattering sensors. For particle sizes 30 µm(c) and larger, data was only obtained from light extinction sensors. Figure 3a shows the results for each participating APC. Participants 5 and 18 exhibit greater deviation from the mean at larger sizes than other participants but were not previously identified as outliers. This suggests that their deviation could be due to their previous 2016 calibration, and not their 2019 ILS calibration. Figure 3b shows the same data represented in a different form. The dotted line illustrates what the relationship between 2016 and 2019 sizes would be if there were no size shift. The solid line shows the actual relationship between the two calibrations based upon the mean particle sizes for each calibration. The dashed lines show the 95 % confidence intervals for the data. The data shows that the small observed shift in particle size is statistically insignificant.

Table 5 — Comparison of 2016 versus 2019 particle sizes based upon ILS results

2016 particle size µm(c)	Number of APC	2019 ILS mean particle size µm(c)	Difference in size %	Standard deviation µm(c)	CV %
1,5	2	1,6	5,35 %	0,09	5,82 %
2	3	2,1	5,36 %	0,20	9,44 %
3	3	3,0	-0,67 %	0,20	6,86 %
4	12	3,9	-1,74 %	0,22	5,54 %
5	16	5,0	-0,57 %	0,27	5,41 %
6	16	6,0	-0,50 %	0,26	4,30 %
7	16	7,0	-0,10 %	0,30	4,24 %
8	16	8,0	0,29 %	0,33	4,09 %
9	16	9,0	0,20 %	0,36	3,98 %
10	16	10,0	-0,05 %	0,40	4,01 %
11	16	11,0	-0,39 %	0,44	4,00 %
12	16	11,9	-1,09 %	0,47	3,96 %
13	16	12,9	-0,63 %	0,54	4,20 %
14	16	14,0	-0,35 %	0,62	4,44 %
15	16	14,9	-0,52 %	0,71	4,75 %
16	16	15,8	-1,37 %	0,80	5,08 %
17	16	16,7	-1,86 %	0,90	5,39 %
18	16	17,6	-2,14 %	0,97	5,53 %
19	16	18,7	-1,66 %	1,08	5,77 %
20	15	19,7	-1,54 %	1,14	5,79 %
21	15	20,8	-0,94 %	1,19	5,73 %
22	15	21,8	-1,13 %	1,21	5,57 %
23	14	22,6	-1,87 %	1,37	6,07 %
24	14	23,4	-2,48 %	1,59	6,80 %
25	13	23,9	-4,20 %	1,27	5,31 %
26	12	24,8	-4,73 %	1,37	5,55 %
27	12	25,5	-5,38 %	1,39	5,44 %
28	12	26,3	-6,05 %	1,45	5,51 %
29	12	27,1	-6,69 %	1,52	5,61 %
30	12	28,0	-6,65 %	1,66	5,94 %
Average			-1,70 %		5,32 %



Key

Y 2019 ILS particle size µm(c)

X ISO 11171:2016 particle size, µm(c)

1 participant 18

2 participant 5

Key

Y 2019 ILS particle size µm(c)

X ISO 11171:2016 particle size, µm(c)

1 no particle size shift (theoretical)

2 ILS mean (actual)

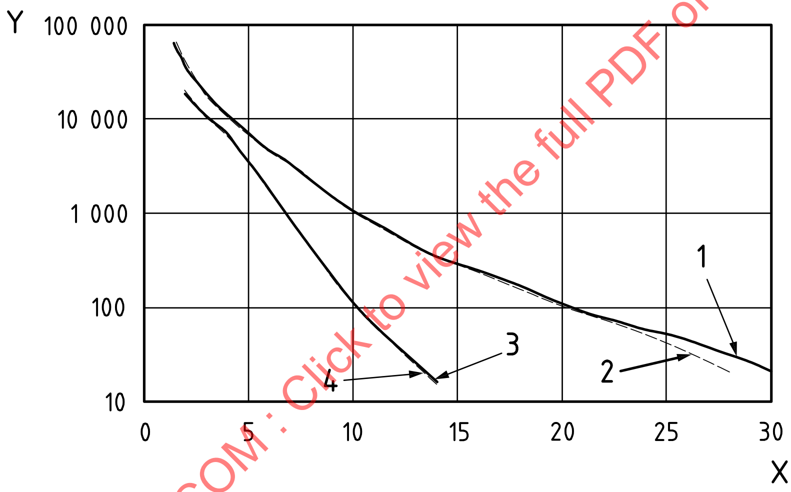
3 95 % confidence levels based upon ILS

Figure 3 — Graphical summary of 2016 versus 2019 particle sizes for ILS APC

To assess the impact of the observed size shift on particle count data, the constrained cubic spline method was applied to the ILS results and used to estimate particle concentration as a function of size for an SRM 2806d candidate sample and an RM 8632a sample using an APC calibrated according to ISO 11171:2016 and according to the 2019 ILS. The particle size distribution of the SRM 2806d candidate sample is comparable to upstream samples collected during an ISO 16889 multi-pass filter test. The size distribution of the RM 8632a sample is comparable to samples collected downstream of a high efficiency filter during a multi-pass filter test. The results are shown in Table 6 and Figure 4. For the SRM 2806d candidate sample, the difference in particle counts between the two calibration methods is less than the ILS reproducibility (refer to Table 2) for all sizes except 2 µm(c) and 30 µm(c). Furthermore, the difference in counts is less than the equivalent of a tenth of an ISO Code unit. On average, the difference is about 5 times lower than the reproducibility, suggesting that the impact of the size shift on particle count data is insignificant. For the RM 8632a samples, the differences in particle counts over the size range from 2 µm(c) to 14 µm(c), the range over which significant counts were obtained, is even smaller compared to the ILS reproducibility (refer to Table 3). These results suggest that ISO 11171:2020 calibration using SRM 2806d will not significantly affect particle count data for the industry, although individual laboratories may detect evidence of a shift due to the uncertainty of their previous ISO 11171:2016 calibration.

Table 6 — Effect of the particle size shift on SRM 2806d candidate sample and RM 8632a sample particle count data

Size, µm(c)	SRM 2806d Candidate Sample			RM 8632a Sample		
	2016 µm(c) Concentration, particles/mL	2019 ILS µm(c) Concentration particles/mL	Difference in concentration %	2016 µm(c) Concentration, particles/mL	2019 ILS µm(c) Concentration particles/mL	Difference in concentration %
2	37 115	40 430	-8,9 %	18 250	19 378	-6,2 %
3	18 237	18 014	1,2 %	10 639	10 528	1,0 %
4	11 175	10 863	2,8 %	6 772	6 561	3,1 %
6	4 685	4 642	0,9 %	1 783	1 761	1,3 %
10	1 073	1 071	0,1 %	113,4	113,2	0,2 %
14	349,7	34,.6	0,9 %	16,2	15,5	4,1%
21	91,0	88,2	3,1 %			
30	21,2	14,0	33,8 %			



Key

Y particle/mL > indicated size

X particle size, µm(c)

1 SRM 2806d candidate, 2016 particle size

2 SRM 2806d candidate, 2019 ILS particle size

3 RM 8632a, 2016 particle size

4 RM 8632a, 2019 ILS particle size

Figure 4 — Effect of the observed particle size shift on particle count data for SRM 2806d candidate

9 Impact of ISO 11171:2020 on filter performance data

To assess the impact of the observed size shift on filter performance results, Beta Ratio versus size data for 5 filters representing a range of performance characteristics was obtained. The Beta Ratio results for each filter as a function of 2016 particle sizes and 2019 sizes are given in Table 7. For illustrative purposes, the results are also shown graphically in Figure 5 along with the results expressed in units of µm(b). The effect of the difference between the 2016 and 2019 µm(c) sizes is negligible for particle sizes 20 µm(c) and smaller. For sizes between about 20 µm(c) and 30 µm(c), the effect is greater, consistent

