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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

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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