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## Impact of changes in ISO fluid power particle counting — Contamination control and filter test standards

*Conséquences des changements survenus dans les normes ISO  
relatives au comptage des particules — Contrôle de la contamination  
et essais de filtres*

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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](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: [Foreword — Supplementary information](#).

The committee responsible for this document is ISO/TC 131, *Fluid power systems*, Subcommittee SC 6, *Contamination control*.

This second edition cancels and replaces the first edition (ISO/TR 16386:1999) which has been technically revised.

## Introduction

This Technical Report has been prepared as an information document to give users an understanding into the background and implications of a number of new and revised contamination control standards, namely ISO 11171, ISO 11943, ISO 16889 and ISO 4406.

The adoption of four revised and updated contamination control standards, ISO 11171, ISO 11943, ISO 16889, and ISO 4406:1999, has produced significant changes in terms of how solid contamination levels and filter performance are reported.

With ISO 11171, the method of calibrating automatic particle counters (APCs) using AC Fine Test Dust (ACFTD) used since the early 1970s has been replaced by a new method traceable to the USA's National Institute of Standards and Technology. As a result, contaminant particle sizes previously referred to as 2  $\mu\text{m}$ , 5  $\mu\text{m}$ , 10  $\mu\text{m}$ , and 15  $\mu\text{m}$  became 4  $\mu\text{m(c)}$ , 6  $\mu\text{m(c)}$ , 10  $\mu\text{m(c)}$ , and 14  $\mu\text{m(c)}$ , respectively, where (c) refers to particle sizing and counting done with an APC calibrated in accordance with ISO 11171.

ISO 11943 is a new standard for calibrating online particle counting systems that are primarily used to evaluate filter performance. With the ISO 16889 filter multi-pass test, which replaces the original ISO 4572 method, ISO Medium Test Dust (ISO MTD) replaces ACFTD as the test dust and the new ISO 11171 traceable particle counter calibration method is used. In ISO 4406:1999, the new calibration method is used, and a new 4  $\mu\text{m(c)}$  size class has been added to the solid contamination code for particle counts made with an automatic particle counter.

These improvements in particle counting and filter testing have a significant impact on contamination control activities. However, it is important to note that there has been no change in the actual contamination levels or in the performance of filters, or their effectiveness in protecting the reliability of components. This Technical Report discusses what the changes are, why they were made, how they impact contamination levels and filter ratings, and how they benefit the industry.

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# Impact of changes in ISO fluid power particle counting — Contamination control and filter test standards

## 1 Scope

This Technical Report discusses the impact of changes in International Standards for particle counting, contamination control, and filter testing.

Liquid automatic particle counters (APCs) are used in monitoring contamination levels in hydraulic fluids, to establish component and assembly cleanliness level specifications, and in determining filter efficiencies and particle size ratings. As a result of the replacement of ISO 4402 with ISO 11171 (APC calibration), the replacement of ISO 4572 with ISO 16889 (multi-pass filter test), and the publication of ISO 11943 (online particle counter calibration), the quality and reliability of particle count and filter test data have improved, increasing their usefulness to industry. However, the resultant redefinition of particle sizes and the use of a new test dust affect how contamination levels and filter performance are reported and interpreted.

NOTE The first editions of ISO 11171, ISO 16889 and ISO 11943 were published in 1999; all three of these International Standards either have been, or are in the process of being, revised.

## 2 Historical background

### 2.1 What is ACFTD?

ACFTD was a test dust that was originally produced in batches by the AC Spark Plug Division of General Motors Corporation. ACFTD was manufactured by collecting dust from a certain location in Arizona (USA), then ball milling and classifying it into a consistent particle size distribution, including particle sizes from roughly 0 µm to 100 µm. The manufacturer supplied the average volumetric particle size distribution of each batch of ACFTD, as determined by either the roller analyser or laser diffraction technique. In 1992, the production of ACFTD ceased.

Because of its relatively consistent particle size distribution, ACFTD had been used to calibrate APCs in ISO 4402 and to evaluate filter performance in ISO 4572 for hydraulic and other applications. With its irregular shape and siliceous nature, ACFTD was believed to be representative of contaminants found in typical hydraulic systems. In ISO 4402, a particle size distribution for ACFTD is given which is based on optical microscopy work done in the late 1960s. At that time, there was no statistical analysis of batch-to-batch variations in ACFTD. Later, it was discovered that differences exist between the published particle size distribution and actual particle size distributions of subsequent batches of ACFTD. These differences are a significant source of variability in particle count results.

### 2.2 Calibrating particle counters using ACFTD

Though often taken for granted, particle counting is the mainstay of contamination control programs. APCs are used to monitor contamination levels in the hydraulic fluid of operating equipment, to establish component and assembly cleanliness level specifications, and to provide a basis for determining filtration ratios (beta ratios), efficiencies, and particle size ratings of hydraulic filters.

Calibration consists of establishing the relationship between APC's threshold voltage setting and particle size. This was done by comparing observed particle contamination levels at known threshold settings to the published ACFTD particle size distribution. Because of this, calibration accuracy depends on the accuracy of the published particle size distribution.

In the absence of a more controlled contaminant, ACFTD had been used for APC calibration for hydraulic and other applications. The ACFTD particle size distribution used for calibration in ISO 4402 is based

on the longest chord dimension of particles as measured by optical microscopy in the late 1960s. At the time, optical microscopy was the most common method used to size and count particles. The goal of the APC calibration procedure was to ensure that particle counts obtained with an APC agreed as closely as possible with counts obtained by optical microscopy.

The accuracy of the published ACFTD particle size distribution and the corresponding APC particle counter calibration has been questioned since the late 1970s. Because the original microscopy work was done on specific batches of ACFTD, the effects of batch-to-batch variability on the particle size distribution and APC calibration were not considered. Despite this, ISO 4402:1991 required laboratories to calibrate to the original published size distribution, even though the particular batch of ACFTD used likely had a different distribution.

### 2.3 The original multi-pass filter test

While the ACFTD method of APC calibration was being developed, the hydraulic filter multi-pass test method was developed to measure filter performance, primarily efficiency and contaminant capacity. In 1981, the multi-pass test was published as ISO 4572:1981 and is still widely used. The characteristics of ACFTD that made it valuable for APC calibration also make it ideal for filter testing. In a multi-pass test, hydraulic fluid is recirculated through the filter under test while a slurry of ACFTD in hydraulic fluid is continually added to a reservoir located upstream of the filter under test. Particle counts are taken both upstream and downstream of the filter under test throughout the test. These counts are used to calculate particle removal efficiency as a function of particle size. The results, expressed as a filtration ratio (beta ratio) depend not only on the APC calibration but also the particle size distribution of the test dust. The retained contaminant capacity of the filter under test is also reported as the amount of ACFTD needed to cause the filter to reach its terminal differential pressure. The particle size distribution and morphology of the test dust also have a significant impact on filter efficiency and retained contaminant capacity.

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## 3 New test dusts

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In 1992, efforts to revise particle counter calibration and filter test standards took on new urgency when the AC Rochester (formerly AC Spark Plug) Division of General Motors Corporation discontinued production of ACFTD. ISO Technical Committee 22 responded by developing ISO 12103-1, a filter test dust standard that specifies the physical, chemical, and particle size distribution characteristics of four silica test dusts. The new test dusts are manufactured by jet milling instead of the ball milling process used for ACFTD. As a result, their particle size distribution and the shape of individual particles differ from ACFTD. Further, ISO 12103-1 specifies electrozone techniques, instead of the roller analyser or laser diffraction methods used in the production of ACFTD, to specify the particle size distribution of the new test dusts. Because of ISO 12103-1, the new test dusts are better controlled, and batch-to-batch variability is less than that of ACFTD.

One of the test dust specified in ISO 12103-1, ISO medium test dust (ISO MTD) (ISO 12103-1 grade A3) was chosen by ISO/TC 131/SC 6 to replace ACFTD for particle counter calibration and multi-pass filter testing. ISO MTD is chemically identical to ACFTD but contains fewer particles smaller than 5 µm and is easier to disperse in hydraulic fluid. The high concentration of fine particles in ACFTD can result in coincidence errors during particle counting. Thus, the use of ISO MTD reduces the potential for error while retaining the desirable characteristics of ACFTD.

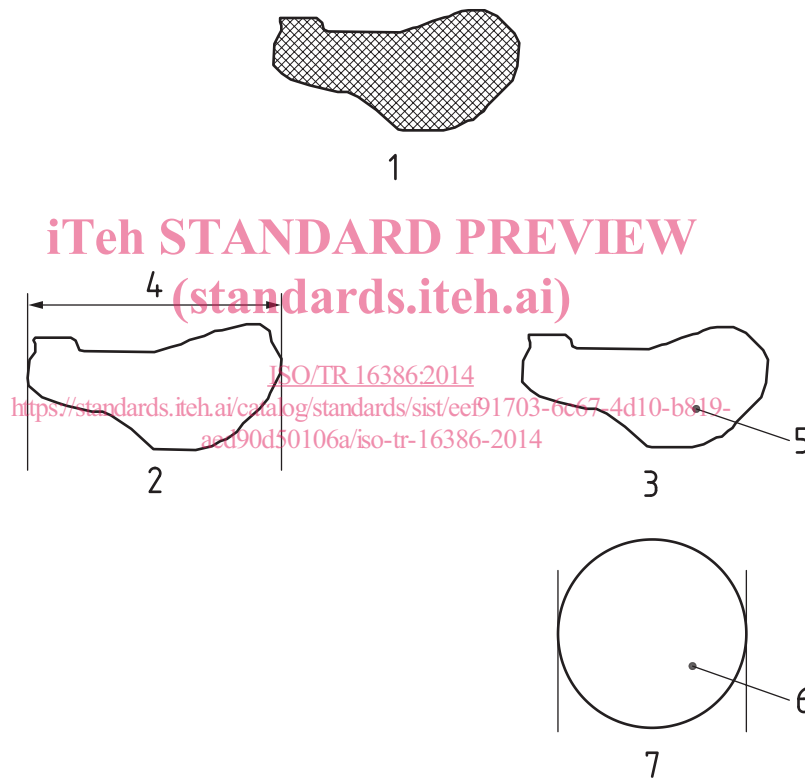
## 4 New APC calibration procedure

Due to concerns about the accuracy of the ACFTD particle size distribution, the National Fluid Power Association (NFPA) in the USA began a project in 1980 to develop a traceable APC calibration method. The first attempt at a traceable method resulted in the American National Standard ANSI/(NFPA) T2.9.6 R1-1990, which used mono-sized latex particles suspended in MIL-H-5606 mineral oil with sizes traceable to the USA's National Institute for Standards and Technology (NIST). Usage of this method was discouraged, however, because shortly after its publication, it was found that different types of APCs calibrated with latex particles showed poor agreement with each other. APCs made by different manufacturers and APCs using different light sources (such as laser diode or white light) or different



measurement principles (light scattering or light extinction) and calibrated using latex particles yielded different particle count results when analysing ACFTD or similar contaminants. This is due to differences in the optical properties of latex and silica. It was concluded that the APC calibration contaminant should be optically similar to the contaminants typically used in filter testing.

In order to develop a traceable particle counter calibration method, NIST was asked in 1993 to certify the particle size distribution of suspensions of particulate contaminant in MIL-H-5606 hydraulic fluid. The certified suspensions, NIST Standard Reference Material (SRM) 2806, consist of 2,8 mg/l suspensions of particulate contaminant in MIL-H-5606 hydraulic fluid. Scanning electron microscopy (SEM) and image analysis software were used to measure the projected area equivalent diameters of particles and to determine the particle size distribution of the SRM. The projected area equivalent diameter is used as the basis for determining particle size because it more closely approximates the dimension actually measured by liquid APCs than the longest chord dimension used to define the ACFTD particle size distribution. A particle sensor measures the change in light intensity caused by the presence of a particle in its sensing zone; in a sense, a light extinction sensor measures the size of the shadow cast by a particle. The difference between the longest chord dimension and the projected area equivalent is illustrated in [Figure 1](#).



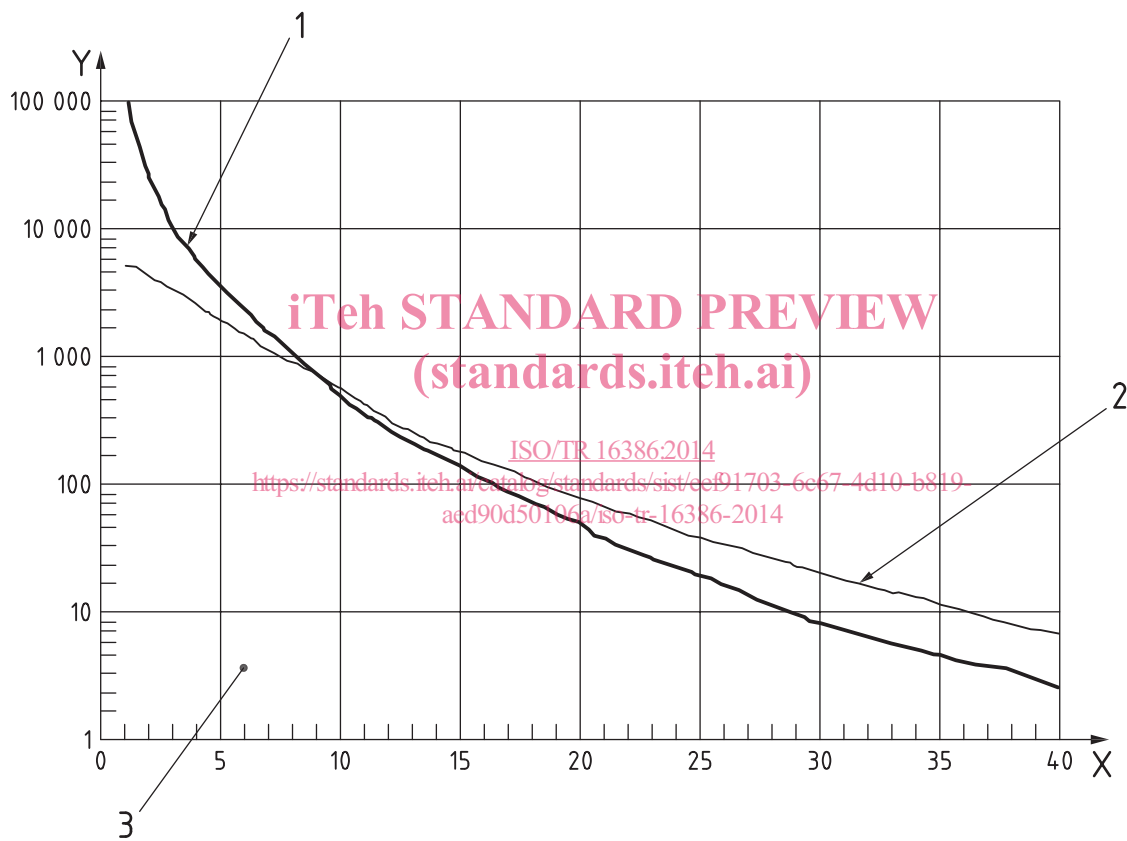
#### Key

- 1 actual size
- 2 as seen by an APC calibrated in accordance with ISO 4402 (ACFTD particle size distribution) - longest chord
- 3 as seen by an APC calibrated using NIST SRM 2806 (NIST particle size distribution) - projected area equivalent diameter
- 4 longest chord dimension  $d = 13 \mu\text{m}$
- 5 area  $= 78,5 \mu\text{m}^2$
- 6 area  $= 78,5 \mu\text{m}^2$
- 7 area equivalent diameter  $d = 10 \mu\text{m}$

**Figure 1 — Particle size ( $d$ ) as defined using longest chord dimension and projected area equivalent diameter**

As shown in Figure 2, particle size distribution data obtained using APCs calibration with the NIST SRM differ considerably from those obtained using an APC calibrated with the ISO 4402 method. The results shown in Figure 2 represent the particle size distribution of 2,8 mg/l suspensions of SAE 5-80 micrometer test dust analysed by NIST and by APCs calibrated using ACFTD in accordance with ISO 4402. The latter results were obtained during an international round robin conducted under the auspices of ISO/TC 131/SC 8. Figure 2 illustrates two interesting features:

- a) at particle sizes smaller than about 10 µm, significantly more particles were observed by APCs calibrated using the NIST SRM than by APCs calibrated using ACFTD. These higher particle counts are a result of the enhanced sensitivity of electron microscopy when compared to optical microscopy;
- b) at particle sizes larger than 10 µm, fewer particles were observed by APCs calibrated using the NIST SRM than by APCs calibrated using ACFTD. This is the case primarily because NIST reported the projected area equivalent diameter of particles, which is smaller than the longest chord dimension used to general the published ACFTD particle size distribution, as illustrated in Figure 1.



**Key**

- X particle size in micrometres
- Y number of particles per millilitre larger than the indicated size
- 1 calibration using NIST SRM 2806
- 2 calibration using ACFTD
- 3 suspension of 2,8 mg/l of SAE 5-80 MICROMETER TEST DUST in hydraulic fluid

**Figure 2 — Comparison of the particle size distribution of a suspension of 2,8 mg/l of SAE 5-80 MICROMETER TEST DUST in hydraulic fluid, measured using the ISO 4402 (ACFTD) and ISO 11171 (NIST SRM 2806) calibration methods**

ISO 11171 (APC calibration), ISO 16889 (multi-pass filter test), and ISO 11943 (online particle counter calibration) all provide traceability to NIST SRM 2806. The quality and reliability of both particle count and filter test data are expected to improve as a result of the use of these International Standards. In 1995, this was established by means of a series of international round robin test programs conducted

to evaluate these International Standards. The results of the round robin test programs were discussed extensively in informative annexes in the first editions of these International Standards.

## 5 Why changes were necessary

ISO and other standards developing organizations are charged with developing technically sound industrial standards that permit valid comparisons among data from different sources. Such standards make it possible to compare data from various suppliers or laboratories that test in accordance with the same standard. For this reason, ISO encourages the use of certified reference materials for calibration. In the past, ACFTD, an uncertified reference material, had been used for both multi-pass filter testing and APC calibration. Although the methods that used ACFTD had long been used to establish a common calibration among laboratories, it had shortcomings that affected the accuracy of the test results and agreement among laboratories. An even greater problem is that ACFTD is no longer commercially available.

From the standpoint of APC calibration, the ISO 4402 calibration method had several inherent weaknesses, perhaps the greatest of which was that the ACFTD particle size distribution was not certified and lacked traceability. Calibration accuracy depends on the accuracy of the reference particle size distribution. For some time, it had been known that modern electron microscopy and electrozone counting techniques yield particle size distributions that differ from the ACFTD particle size distribution given in ISO 4402, which were obtained by optical microscopy, particularly for particles smaller than about 10  $\mu\text{m}$ . The ACFTD particle size distribution given in ISO 4402 was based on analyses done in the late 1960s on specific batches of ACFTD and ignored batch-to-batch variability. Further, the ISO 4402 calibration method does not require validation of the APC or qualification of the analytical techniques used. The ISO 11171 calibration method corrects many of these deficiencies through the use of certified calibration suspensions. In addition, it establishes minimum APC performance requirements and uses statistical methods to evaluate the data and analytical techniques. One consequence of the change in calibration method is a redefinition of particle sizes, as discussed later in this Technical Report.

From the standpoint of filter testing, the changes to a new test dust and APC calibration method for multi-pass filter testing were necessitated by the lack of commercial availability of ACFTD. As had been mentioned previously, the replacement of ACFTD with ISO MTD offered several advantages for filter testing:

- a) compared with ACFTD, ISO MTD is more reproducible in its properties and particle size distribution, is more defined, and is easier to disperse in hydraulic fluid;
- b) ISO MTD has significantly fewer particles smaller than 5  $\mu\text{m}$ , which reduces the risk of coincidence errors in particle counting; this is particularly important in the online particle counting systems used in multi-pass testing.

In addition to the change in test dust, improvements in the multi-pass testing and reporting techniques have been included in ISO 16889. Unfortunately, the differences in the particle size distributions and particle morphology of ACFTD and ISO MTD, as well as the changes in testing and reporting techniques, contribute to differences between results from tests conducted in accordance with ISO 4572 and ISO 16889. However, ISO 16889 provides an overall improvement in the repeatability of multi-pass filter test results; the impact of this is discussed later in this Technical Report.

## 6 Impact on particle sizes and contamination measurements

### 6.1 Redefinition of particle sizes

Both particle size and concentration are important in contamination control. The change in APC calibration method results in an immediate change in reported particle sizes and concentrations. The magnitude of the change depends on the particle size of interest. Because of this change, ISO 11171 specifies that particle sizes measured by an APC calibrated in accordance with that International Standard should be expressed in the unit  $\mu\text{m}(\text{c})$ , where the (c) refers to calibration in accordance with ISO 11171. [Table 1](#) compares particle sizes measured by APCs calibrated in accordance with ISO 4402 and ISO 11171. At the particle size 10  $\mu\text{m}$ , there is only about a 2 % difference in the reported size. However, the ISO 4402 particle size of 15  $\mu\text{m}$  determined using an APC calibrated with ACFTD in accordance