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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

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this Technical Report may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TR 16386 was prepared by Technical Committee ISO/TC 131, *Fluid power systems*, Subcommittee SC 6, *Contamination control and hydraulic fluids*.

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, ISO 11171, ISO 11943, ISO 16889 and ISO 4406:1999.

Introduction

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 AC Fine Test Dust (ACFTD) particle counter calibration method used since the early 1970s has been replaced with a new National Institute of Standards and Technology (NIST) traceable particle counter calibration method. As a result, contaminant sizes previously referred to as 2 µm, 5 µm, 10 µm and 15 µm, will become approximately 4,6 µm(c); 6,4 µm(c); 9,8 µm(c) and 13,6 µm(c), respectively, where (c) refers to calibration per ISO 11171. ISO 11943 is a new standard for calibrating on-line particle counters, primarily used to evaluate filter performance. With the ISO 16889 multi-pass filter test, which replaces the original ISO 4572 method, ISO Medium Test Dust (ISO MTD) replaces ACFTD as the test dust and the new traceable particle counter calibration method is used. In ISO 4406:1999, the new calibration method is used and a new 4 µm(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 nor in the performance of filters, or their effectiveness in protecting the reliability of components. This report discusses what the changes are, why they have been made, how they will impact contamination levels and filter ratings, and how they benefit the industry.

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

1 Scope

Liquid automatic optical particle counters (APCs) are used in monitoring contamination levels in hydraulic oil, to establish component and assembly cleanliness level specifications, and in determining filter efficiencies and size ratings. As a result of the replacement of ISO 4402 with ISO 11171 (particle counter calibration), the replacement of ISO 4572 with ISO 16889 (multi-pass filter test), and the new ISO 11943 (on-line particle counter calibration), it is anticipated that the quality and reliability of particle count and filter test data will improve, increasing their usefulness to industry. However, the resultant redefinition of particle sizes and the use of a new test dust affects how contamination levels and filter performance are reported and interpreted. The impact of these changes is discussed in this report.

2 Historical background

2.1 What is ACFTD?

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

Because of its relatively consistent particle size distribution, ACFTD has 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 number size distribution for ACFTD is given that 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 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. Automatic particle counters are used to monitor contamination levels in the oil of operating equipment, to establish component and assembly cleanliness level specifications, and to provide a basis for determining filter beta ratios, efficiencies and size ratings.

Calibration consists of establishing the relationship between APC threshold voltage setting and particle size. This was done by comparing observed particle concentrations at known threshold settings to the published ACFTD size distribution. Calibration accuracy is dependent on the accuracy of the published size distribution.

In the absence of a more controlled contaminant, ACFTD has been used for APC calibration for fluid power and many 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 obtain particle counts. 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 size distribution, and the corresponding APC particle counter calibration, has been questioned since the late 1970s. Since the original microscopy work was done on specific batches of ACFTD, the effects of batch to batch variability on the size distribution and APC calibration were not considered. Despite this, ISO 4402:1991 requires laboratories to calibrate to the original published size distribution, even though the particular batch of ACFTD being used may differ.

2.3 The original multi-pass filter test

While the ACFTD method of particle counter calibration was being developed, the hydraulic filter multi-pass test method was developed to measure filter performance, primarily efficiency and dirt capacity. In 1981, the multi-pass test was adopted as an International Standard, 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, oil is recirculated through a test filter while a slurry of ACFTD is continually added to a reservoir located upstream of the filter. Particle counts are taken, both upstream and downstream of the filter, throughout the test. These are used to calculate particle removal efficiency as a function of particle size. The results, expressed as filtration or beta ratios, are obviously dependent on the APC calibration, but also on the particle size distribution of the test dust. The retained dirt capacity of the test filter 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 dirt capacity.

3 New test dusts

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 TC 22 responded by adopting 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 using 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 analyzer or laser diffraction methods used in the production of ACFTD, to specify the particle size distribution of the new dusts. As a result of ISO 12103-1, the new test dusts are better controlled and batch-to-batch variability is less than with the old ACFTD.

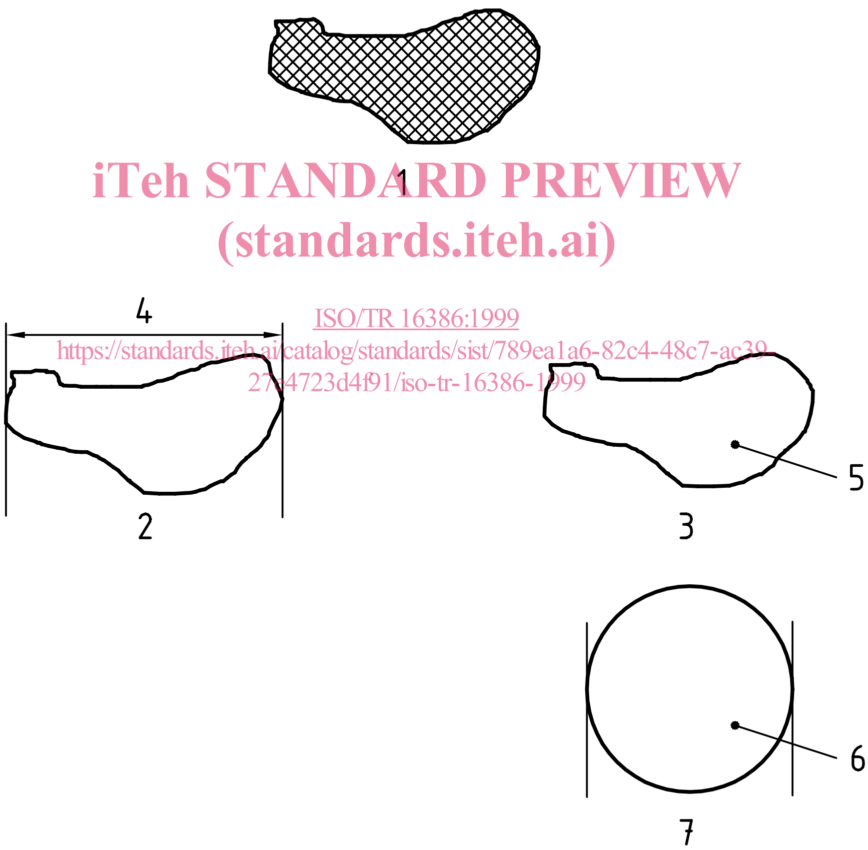
One of the dusts described in ISO 12103-1, ISO Medium Test Dust (grade A3) , was chosen by ISO/TC 131/SC 6 to replace ACFTD for particle counter calibration and multi-pass filter testing. ISO MTD is physically and chemically identical to ACFTD, but contains fewer particles smaller than 5 µm and is easier to disperse in oil. The high concentration of fine particles in ACFTD can result in coincidence errors when particle counting. Thus, the use of ISO MTD reduces this source of 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 (USA) began a project in 1980 to develop a traceable APC calibration method. The first attempt at a traceable method resulted in a new method, USA standard ANSI/(NFPA) T2.9.6R1 (1990), that used mono-sized latex particles suspended in MIL-H-5606 mineral oil with sizes traceable to NIST. Usage of this method has been discouraged, however, because shortly after its introduction, it was found that poor agreement was obtained between different types of APCs calibrated with latex. APCs made by different manufacturers and APCs utilizing different light sources (such as laser diode or white light) or different measurement principles (light scattering or light extinction) yielded different particle count results when analyzing ACFTD or similar samples. 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 ISO MTD. The certified suspensions, NIST Standard Reference Material (SRM) 2806, consist of 2,8 mg/L suspensions of ISO MTD in Mil-H-5606 hydraulic fluid. Scanning electron microscopy and statistical analysis techniques were used to measure the projected area equivalent diameters of ISO MTD 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 automatic optical particle counters than the longest chord dimension used to define the ACFTD 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 projected area equivalent diameter is illustrated in Figure 1.

As shown in Figure 2 for ISO MTD, size distribution results obtained using the NIST calibration differ considerably from those obtained using an APC calibrated with the ACFTD calibration method. The results shown in the figure represent the particle size distribution results for 2,8 mg/L samples of ISO MTD analyzed by NIST and by particle counters using the ACFTD calibration. The latter results were obtained by an international round robin conducted under the auspices of ISO TC 131/SC 8/WG 9. Two interesting features are apparent from the figure. At sizes below about 10 µm, significantly more particles were observed by NIST than with the ACFTD calibration. The higher particle counts are a result of the enhanced sensitivity of scanning electron microscopy when compared to optical microscopy. At sizes larger than 10 µm, fewer particles were observed by NIST. This is primarily because NIST reported the projected area equivalent diameter of particles, which is smaller than the longest chord dimension used to generate the published ACFTD size distribution (refer to Figure 1).



Key

1 Actual size

2 As seen by ISO 4402 (ACFTD size distribution)

3 As seen by NIST (NIST size distribution) Projected area equivalent diameter

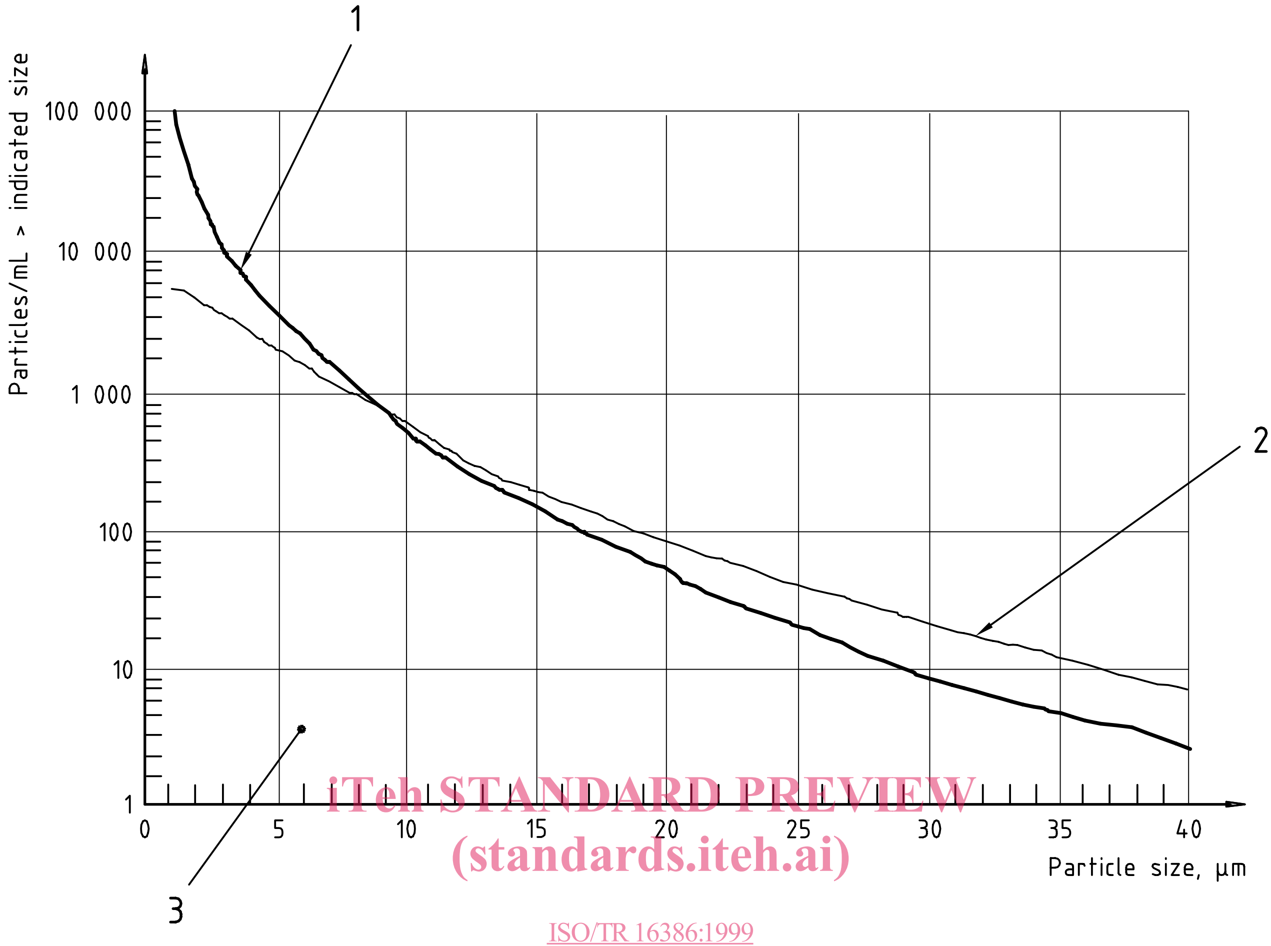
4 Longest chord dimension d = 13 µm

5 Area = 78,5 µm^2

6 Area = 78,5 µm^2

7 Area equivalent diameter d = 10 µm

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



Key

1 NIST Calibration

2 ACFTD Calibration

3 2,8 mg/L of ISO Medium Test Dust

Figure 2 — Comparison of ISO Medium Test Dust size distribution as measured using the ACFTD (ISO 4402) and the new NIST traceable (ISO 11171) calibration methods.

The revisions to ISO 4402 (particle counter calibration), ISO 4572 (multi-pass filter test), and ISO 11943 (on-line particle counter calibration) all utilize ISO MTD and provide traceability to NIST SRM 2806. It is anticipated that the quality and reliability of both particle count and filter test data will improve, as a result. In 1995, this was established by means of a series of international round robins conducted to evaluate these revisions. The results of the round robins are discussed extensively in informative annexes in the respective individual ISO standards.

5 Why changes are necessary

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

From the standpoint of APC calibration, the ACFTD calibration method has several inherent weaknesses, perhaps the greatest of which is that the ACFTD size distribution is uncertified and lacks traceability. Calibration accuracy depends on the accuracy of the reference particle size distribution. For some time, it has been known that modern electron microscopy and electrozone counting techniques yield size distributions at variance with the published (ISO 4402) ACFTD size distribution obtained by optical microscopy, particularly for particles smaller than about 10 µm. The published ACFTD size distribution is based on analyses done in the late 1960s on specific batches of ACFTD and ignores batch-to-batch variability. Further, the ACFTD method does not require validation of the APC or qualification of the analytical techniques used. The new 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, which will be discussed later in this report.

From the standpoint of filter testing, the changes to a new test dust and calibration method for multi-pass filter testing are necessitated by the lack of commercial availability of ACFTD. As has been mentioned previously, the replacement of ACFTD with ISO MTD offers several advantages for filter testing. ISO MTD is more reproducible in its properties and size distribution, is better specified, and easier to disperse than ACFTD. ISO MTD has significantly fewer particles smaller than 5 µm. This reduces the risk of coincidence errors in particle counting. This is particularly important for the on-line particle counting systems used in multi-pass testing. In addition to the change in test dust, improvements in the multi-pass test and reporting techniques have been included in ISO 16889. Unfortunately, the differences in the size distributions and particle morphology of ACFTD and ISO MTD, as well as the technique and reporting changes, will contribute to differences in multi-pass filter test results when compared to those from ISO 4572:1981. However, there should be an overall improvement in the repeatability of multi-pass test results. The impact of this is discussed later.

6 Impact on particle sizes and contamination measurements

6.1 Redefinition of particle sizes

Both particle size and concentration are important in contamination control. With the change in calibration method, there will be 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 should be written using the symbol µm(c), where (c) refers to calibration per ISO 11171. Table 1 compares ACFTD sizes to the new NIST sizes. At 10 µm, there is only about a 2% difference in the reported size. However, the ACFTD 15 µm will become about 13,6 µm(c), while the old 5 µm will become about 6,4 µm(c) with the NIST traceable method. The biggest change occurs at very small particle sizes. Particles reported as 1 µm in size with the ACFTD method will become about 4,2 µm(c). This change in reported size can lead to confusion when reporting contamination levels or comparing filter beta ratios and efficiencies.

Table 1 — Comparing Particle Sizes Obtained by Different Calibration Methods

To convert from ACFTD size to NIST size		To convert from NIST size to ACFTD size	
an ACFTD size of: (ISO 4402:1991) µm	corresponds to a NIST size of: (ISO 11171) µm(c)	a NIST size of: (ISO 11171) µm(c)	corresponds to an ACFTD size of: (ISO 4402:1991) µm
1	4,2	4	< 1
2	4,6	5	2,7
3	5,1	6	4,3
5	6,4	7	5,9
7	7,7	8	7,4
10	9,8	9	8,9
15	13,6	10	10,2
20	17,5	15	16,9
25	21,2	20	23,4
30	24,9	25	30,1
40	31,7	30	37,3

