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Hydraulic fluid power — Determination of the particulate contamination level of a liquid sample by automatic particle counting using the light-extinction principle

Transmissions hydrauliques — Détermination du niveau de pollution particulaire d'un échantillon liquide par comptage automatique des particules par absorption de lumière

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

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.

ISO 11500 was prepared by Technical Committee ISO/TC 131, *Fluid power systems*, Subcommittee SC 6, *Contamination control*.

This second edition cancels and replaces the first edition (ISO 11500:1997), which has been technically revised, specifically with the following major differences:

a) inclusion of a “quick check” method to determine the presence of water in the sample;

b) update of the method for calibrating the automatic particle counter (APC) from ISO 4402[1)] to ISO 11171;

c) elimination of requirement to analyse samples in a class 100 000 clean room;

d) improved dilution methods;

e) improved guidelines on APC operation, including guidance for detecting and overcoming coincidence error;

f) revision of how to check the validity of the reported particle count.

It also incorporates the Technical Corrigendum ISO 11500:1997/Cor. 1:1998.

1) ISO 4402, *Hydraulic fluid power — Calibration of automatic-count instruments for particles suspended in liquids — Method using AC Fine Test Dust contaminant*. Withdrawn and revised by ISO 11171.

Introduction

In hydraulic fluid power systems, power is transmitted and controlled through a liquid under pressure within an enclosed circuit. The liquid is both a lubricant and a power-transmitting medium.

The presence of solid contaminant particles in the liquid interferes with the ability of the hydraulic fluid to lubricate and causes wear to the components. The extent of contamination in the fluid has a direct bearing on the performance and reliability of the system and it is necessary to control solid contaminant particles to levels that are considered appropriate for the system concerned.

A quantitative determination of particulate contamination requires precision in obtaining the sample and in determining the extent of contamination. The liquid automatic particle counter (APC), which works on the light-extinction principle, has become an accepted means of determining the extent of contamination. The accuracy of particle count data can be affected by the techniques used to obtain such data.

This International Standard details procedures for the analysis of contaminated liquid samples using an automatic particle counter. Correct use of an automatic particle counter helps to reduce errors and enhances the accuracy of reproducibility in data.

Hydraulic fluid power — Determination of the particulate contamination level of a liquid sample by automatic particle counting using the light-extinction principle

1 Scope

This International Standard specifies an automatic particle counting procedure for determining the number and sizes of particles present in hydraulic-fluid bottle samples of clear, homogeneous, single-phase liquids using an automatic particle counter (APC) that works on the light-extinction principle.

This International Standard is applicable to the monitoring of

a) the cleanliness level of fluids circulating in hydraulic systems,

b) the progress of a flushing operation,

c) the cleanliness level of support equipment and test rigs,

d) the cleanliness level of packaged stock.

NOTE 1 Measurements can be made with particles suspended in the original liquid or in a sample of the liquid diluted with a compatible liquid to reduce coincidence error.

NOTE 2 The presence of a fluid interface obstructs the light beam and gives false signals.

2 Normative references

The following referenced documents are indispensable for the application 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 3722, *Hydraulic fluid power — Fluid sample containers — Qualifying and controlling cleaning methods*

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

ISO 5598, *Fluid power systems and components — Vocabulary*

ISO 11171:1999, *Hydraulic fluid power — Calibration of automatic particle counters for liquids*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5598 and ISO 11171:1999 and the following apply.

3.1
agglomerate
two or more particles that are in intimate contact and cannot be separated by gentle stirring and the small shear forces thus generated

3.2
coincidence error limit
highest concentration of ISO ultrafine test dust (ISO 12103-1, A1, or ISO UFTD) that can be counted with an automatic particle counter with less than 5 % error resulting from the presence of more than one particle in the sensing volume at a time

[ISO 11171:1999, 3.4]

3.3
light extinction
reduction in intensity of a light beam passing through the sensing volume caused by the interaction of the light with single particles

NOTE This is also known as light blockage or light obscuration.

3.4
sensing volume
portion of the illuminated region of the sensor through which the fluid stream passes and from which the light is collected by the optical system

[ISO 11171:1999, 3.2]

3.5
threshold noise level
minimum voltage setting of the particle counter at which the observed pulse-counting frequency does not exceed 60 counts/min due to electrical noise

[ISO 11171:1999, 3.1]

4 Materials and equipment

4.1 Automatic particle counter (APC) for liquids, based on the light-extinction principle, consisting of an instrument to size and record the electrical signals generated when a single particle passes through a sensor for counting within the specified range.

The APC shall be capable of cumulative counting. The APC shall include an automatic bottle-sampling apparatus or a means of moving the sample liquid through the sensor and then into a calibrated volumetric measuring device without altering the particle size distribution of the sample.

4.2 Bottle sampler, used to transfer the liquid being analysed through a sensor, which may be an auxiliary component or a part of the APC itself.

If gas is used to force the liquid through the sensor, the gas shall be filtered through a 0,45 µm filter and shall be free from oil and water.

4.3 Density meter, with an accuracy of 0,001 g/cm^3, if the mass-dilution method is used.

4.4 Electronic balance, calibrated, with a resolution of 0,1 mg or better.

4.5 Hot plate, capable of heating to 150 °C ± 2 °C.

4.6 Sample-agitating device, such as a **laboratory bottle roller** or **paint shaker**, capable of re-dispersing the contaminant in the liquid sample without altering the basic particle size distribution of the contaminant.

4.7 Sample bottles, normally cylindrical bottles made of glass or high-density polyethylene, fitted with either a non-shedding, threaded cap forming a seal with the bottle without the use of an insert, or a cap with an internal seal.

The dimensions of the bottle depend upon the type of sample-bottle facility in use with the APC, but bottles should normally have a capacity of 250 mL. The bottle should be flat-bottomed and wide-necked to facilitate cleaning. The sample bottles shall be cleaned and verified in accordance with ISO 3722.

NOTE It has been found that a cleanliness level of fewer than 10 particles > 4 µm(c) and fewer than 2 particles > 6 µm(c) per millilitre of sample-bottle volume is adequate.

4.8 Liquid dispensers, fitted with a 0,45 µm membrane filter directly at the outlet.

4.9 Temperature-measuring device, calibrated, with an accuracy of ± 1 °C or better.

4.10 Timer, capable of measuring minutes and seconds, calibrated, with an accuracy of 0,1 s or better.

4.11 Ultrasonic bath, rated at a power intensity of 3 000 W/m^2 to 11 000 W/m^2 of the base area, which has been shown to be an acceptable means of both dispersing agglomerated particles within the liquid and removing air introduced by manual agitation.

4.12 Volumetric glassware, consisting of a range of calibrated graduated cylinders and graduated syringes or dosing pipettes (standard total displacement volumetric ware with multiple markings or air displacement) conforming to an appropriate standard and cleaned and verified in accordance with 6.2.

NOTE Examples of appropriate standards for volumetric glassware include ISO 4788 and ISO 8655 (all parts).

5 Diluent liquid

NOTE Observe standard laboratory safety practices and regulations when handling diluents.

5.1 The diluent liquid shall be cleaned to contain fewer than 15 particles > 4 µm(c) per millilitre.

5.2 The diluent liquid shall be physically and chemically compatible with both the sample liquid and the apparatus used.

NOTE See Annex B for examples.

5.3 Care shall be taken to ensure that the diluent does not affect the particle counts.

NOTE See Annex C for information about a method for pre-cleaning the diluent.

6 Pre-test requirements and procedures

6.1 Precautions

6.1.1 Chemicals

Good laboratory practices should be observed in the preparation and use of chemicals used in these procedures, as they can be harmful, toxic or flammable. Take care to ensure compatibility of the chemicals with the materials used. Refer to the material safety data sheet (MSDS) for each chemical and follow the precautions for safe handling and usage described therein.

6.1.2 Electrical interference

Precautions should be taken to ensure that the test area does not exceed the radio-frequency interference (RFI) and electro-magnetic interference (EMI) capabilities of the APC.

NOTE 1 An APC is typically a high-sensitivity device and can be affected by RFI or EMI.

The voltage supply to the instrument shall be stable and free of electrical noise.

NOTE 2 The use of a constant-voltage transformer is considered appropriate.

6.1.3 Use of magnetic stirrer

Do not use a magnetic stirrer for samples containing ferrous or other magnetic particles. If such a stirrer is fitted as standard equipment, it can be necessary to remove or negate the drive magnet.

6.1.4 Relative humidity

The relative humidity of the test area should be controlled within the range of 40 % RH to 70 % RH.

NOTE Relative humidity can affect the particle counts.

6.1.5 Sample storage

Store samples susceptible to bacterial growth under refrigerated conditions (at 5 °C ± 2 °C). Bring refrigerated samples to room temperature prior to evaluation and analyse within 1 h of reaching room temperature.

6.2 Glassware cleaning procedure

6.2.1 Clean all glassware using a validated cleaning procedure. Validate cleanliness in accordance with ISO 3722. The final solvent liquid used for the flush should be

a) filtered petroleum spirit or equivalent, if the samples being analysed are petroleum-based or synthetic liquids,

b) either propan-2-ol (2-propanol) or filtered demineralized water, if the samples being analysed are water-based liquids.

6.2.2 The required cleanliness level (RCL) of all glassware shall be such that contaminant thereon cannot significantly contribute to the overall result.

NOTE It has been found that a cleanliness level of fewer than 10 particles > 4 µm(c) and fewer than 2 particles > 6 µm(c) per millilitre of the glassware's volume is adequate.

6.2.3 Filter all liquids used for cleaning and rinsing through a 1 µm or finer membrane filter.

6.3 APC calibration procedure

Calibration of the APC shall be maintained in accordance with ISO 11171.

6.4 APC operation

6.4.1 Use the APC in accordance with its manufacturer's instructions. Make all measurements at particle concentrations that are below 80 % of the manufacturer's stated coincidence error limit (see 3.2) and at a particle size that is at least 1,5 times above the threshold noise level (see 3.5) of the instrument.

6.4.2 Ensure that the APC has been switched on for long enough to become stabilized.

6.4.3 Clean the sensor and associated pipework prior to use by flushing them with filtered solvent (see 6.2.1).

NOTE This cleaning can be achieved by filling a clean sample bottle with filtered solvent and flushing the solvent through the sensor and associated pipework at a flow rate that is approximately 50 % higher than the flow rate used during analysis.

Ensure that the sampling probe is dried before analysing a sample, otherwise errors can result from the creation of an optical interface between the liquids.

6.4.4 If the sensor has been used previously to analyse a liquid that is not miscible with the liquid being analysed, clean the sensor using the cleaning procedure in 7.4.

6.4.5 Inspect the sensing volume on a regular basis for the presence of particles either in the sensing volume itself or in the entry to the sensing volume.

6.4.6 Verify the cleanliness level of the particle-counting system, including the APC, diluent and glassware, by analysing a volume of filtered diluent that conforms to the requirements given in Clause 5.

6.5 Sample inspection and preparation before counting

6.5.1 Outline

See Figure 1 for a flowchart that illustrates the procedure for the preparation of a liquid sample for automatic particle counting.

6.5.2 Initial preparation and inspection

Remove any visible contamination from the exterior of the closed sample bottle using a lint-free cloth, and visually inspect the sample for

a) cloudiness (which can be an indication of excessive particles or free water),

b) macroscopic particles,

c) free water,

d) inappropriate containers (i.e. leaking or damaged containers or containers not conforming to 4.7),

e) excessive volume (i.e. the sample fills more than 80 % of the sample bottle volume).

A sample exhibiting the phenomena described in items a) through d) shall not be counted using the method specified in this International Standard, as these conditions are likely to affect the performance of the sensor. Record the results of visual inspection in the test report [see Clause 8, item p)].

If sample volume is excessive, proceed to 6.5.3.

If the sample does not exhibit items a) through d), proceed to 6.5.4.

While this International Standard does not require a microscopic examination of the sample, one may be conducted, if desired.

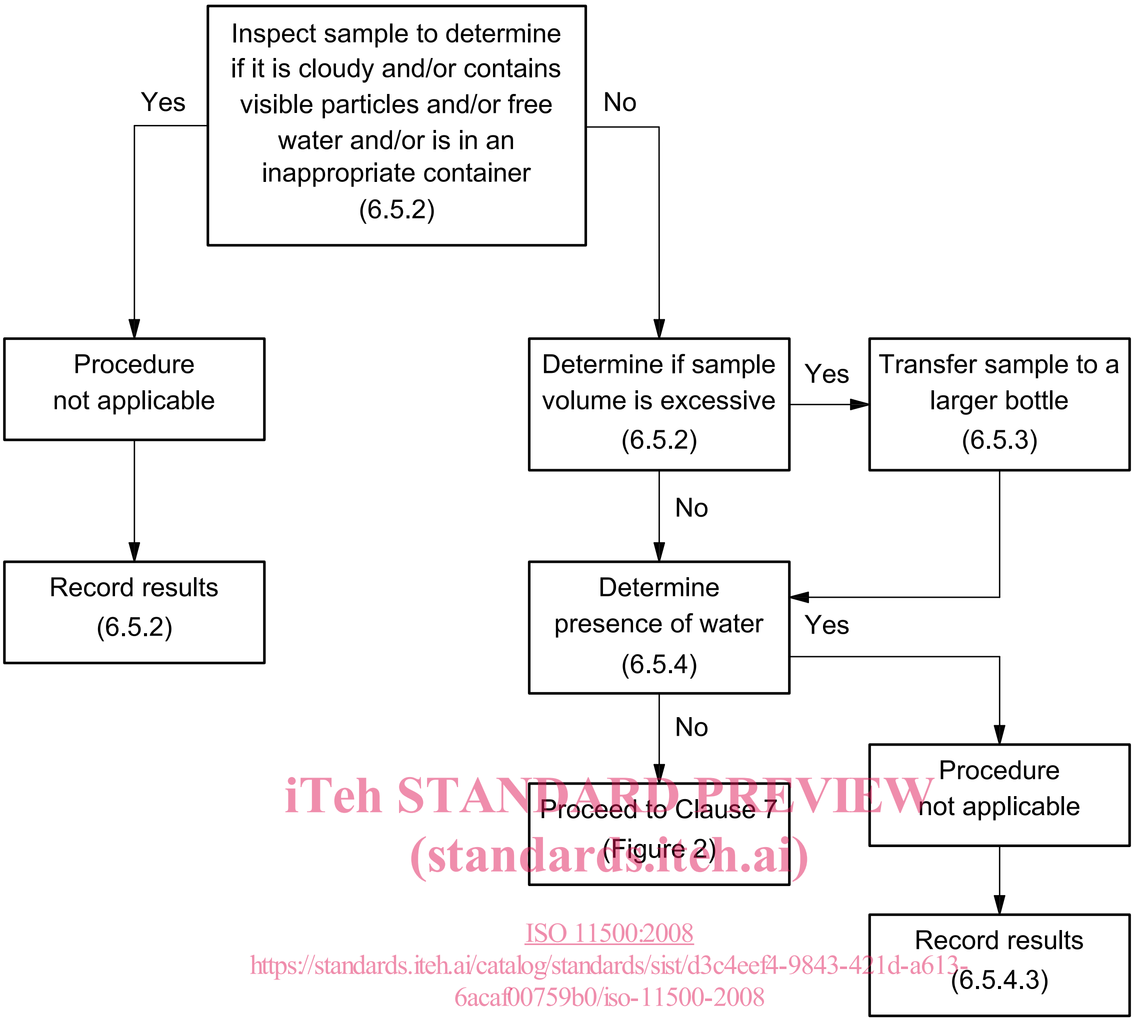

Figure 1 — Flowchart for inspection and preparation of a liquid sample before automatic particle counting

6.5.3 Preparation of sample of excessive volume

6.5.3.1 Estimate the fluid volume in the sample bottle. If the sample takes up less than 80 % of the bottle volume, it may be used. If it takes up more than 80 % of the bottle volume, reduce the volume, following the procedure given in 6.5.3.2 through 6.5.3.4.

NOTE It is difficult to achieve re-dispersion of particulate contaminant in sample bottles that are filled to more than 80 % of the bottle volume, which results in a non-homogenous suspension. Use of the procedure specified in 6.5.3.2 and 6.5.3.4 corrects this problem.

Use caution to ensure that contamination is not added by using the procedure in 6.5.3.2 and 6.5.3.4.

6.5.3.2 Estimate the volume of the sample and select a clean sample bottle (secondary bottle) such that it is about 50 % to 80 % full when the complete sample is transferred into it. This bottle should fit into the sample-agitating device and the bottle-sampling apparatus.

6.5.3.3 Transfer the sample into the secondary sample bottle as follows.

a) Pour approximately half of the sample into the secondary bottle.

b) Vigorously shake the remaining sample by hand.

c) Immediately pour the remaining sample into the secondary sample bottle.

