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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 particles par absorption de lumière

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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).

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*.

This third edition cancels and replaces the second edition (ISO 11500:2008), which has been technically revised.

The main changes are as follows:

— the cleanliness requirements for sample containers have been updated to match ISO 11171;

— the instructions for flushing and diluting solutions using propan-2-ol (2-propanol) and demineralised water have been removed;

— Annex E has been deleted.

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

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 essential to control solid contaminant particles to levels that are considered appropriate for the system concerned.

A quantitative determination of particulate contamination involves 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 document 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 document 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 document 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 fluid.

NOTE Measurements can be made with particles suspended in the original liquid or in a sample of the liquid diluted with a compatible liquid when APC coincidence error limits are exceeded.

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 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 11171:2022, *Hydraulic fluid power — Calibration of automatic particle counters for liquids*

ASTM E694-18, *Standard specification for laboratory glass volumetric apparatus*

3 Terms and definitions

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

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

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

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

3.1
dilution factor
state when the liquid sample is diluted, at which the multiplier coefficient is required to calculate the number concentration of particles contained in the original liquid sample

3.2
dilution ratio
ratio of the amount of diluted liquid sample to the amount of the diluents added at the time of dilution

3.3
coincidence error limit
highest concentration of NIST RM 8632x that can be counted with an *automatic particle counter* (3.7) with an error of less than 5 % resulting from the presence of more than one particle in the *sensing volume* (3.5) at one time

[SOURCE: ISO 11171:2022, 3.5]

3.4
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 1 to entry: This is also known as light blockage or light obscuration.

3.5
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

[SOURCE: ISO 11171:2022, 3.3]

3.6
threshold noise level
minimum voltage setting of an *automatic particle counter* (3.7) at which the observed pulse-counting frequency does not exceed 60 counts/min due to electrical noise in the absence of flow in the *sensing volume* (3.5)

[SOURCE: ISO 11171:2022, 3.2]

3.7
automatic particle counter
APC
instrument that automatically:

a) senses individual particles suspended in a controlled volume of fluid using optical *light extinction* (3.4) or light scattering principles;

b) measures the size of particles;

c) sorts or compiles particles into size ranges;

d) counts particles in each size range;

e) reports the number of particles in each size range per unit volume; and

f) facilitates instrument calibration according to this document

[SOURCE: ISO 11171:2022, 3.1, modified — Note 1 to entry has been deleted.]

4 Materials and equipment

4.1 Automatic particle counter (APC) for liquids, with bottle sampler utilizing the light extinction principle with at least 8 channels that can be sent at various threshold settings calibrated in accordance with ISO 11171.

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 Mechanical shaker, such as a paint or laboratory shaker, suitable for dispersing suspensions.

4.7 Sample bottles, normally flat-bottomed cylindrical bottles made of glass or high-density polyethylene fitted with closures (appropriate bottle caps, for example), and with cleanliness levels lower than 0,5 % of the number of particles (larger than the smallest particle size of interest) expected to be observed in the samples, confirmed in accordance with ISO 3722.

The dimensions of the bottle depend upon the type of bottle sampler in use with the APC, but bottles should normally have a minimum capacity of 250 ml.

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 bottom area.

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 at least Class B, as defined in ASTM E694-18. The glassware shall be 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

IMPORTANT — Observe standard laboratory safety practices when handling diluents.

5.1 The diluent liquid shall be cleaned to cleanliness levels lower than 0,5 % of the number of concentration of particles (larger than the smallest particle size of interest), which are expected to be observed in the samples, confirmed in accordance with ISO 3722.

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

5.2 The diluent liquid shall be physically and chemically compatible with both the sample liquid and the apparatus used. The diluent is considered acceptable if its refractive index is similar to the refractive index of the hydraulic liquid sample. This is to ensure that the diluent does not affect the particle counts.

The use of a volatile solvent can cause erroneous particle counts due to the generation of air bubbles, i.e. “boiling”, if its vapour pressure is reached in the sensor. This can be alleviated by ensuring that the pressure in the sensor is always above the vapour pressure of any liquids used.

It is necessary to take care when diluting to ensure that the sample and the diluent are mutually miscible and to ensure that the additive packages of the sample oil and diluent are compatible.

NOTE See Annex B for examples.

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

Determining the threshold noise level of the APC should be completed in accordance with ISO 11171:2022, A.2. Precautions should be taken to ensure that the threshold noise level is determined with all potential sources of radio frequency interference (RFI) and electro-mechanical interference (EMI) active to simulate the end use case.

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

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

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 eliminate 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 outside of the prescribed range can affect the particle counts.

6.1.5 Sample storage

Store samples susceptible to bacterial growth in sealed containers under refrigerated conditions (at 5 °C ± 2 °C). Bring refrigerated samples to room temperature prior to unsealing to avoid condensation being introduced. Evaluation and analysis shall be completed 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 filtered petroleum spirit or equivalent, if the samples being analysed are petroleum-based or synthetic 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.

The glassware shall be cleaned to cleanliness levels lower than 0,5 % of the number of concentration of particles (larger than the smallest particle size of interest), which are expected to be observed in the samples, confirmed in accordance with ISO 3722.

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 within the operating limits previously determined by ISO 11171 (refer to 6.3).

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 plumbing lines prior to use by flushing them with filtered solvent (see 6.2.1).

NOTE Cleaning can be achieved by filling a clean sample bottle with filtered solvent, and then flushing the solvent through the sensor and associated plumbing lines, 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 7.3.10.

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).

A sample exhibiting the phenomena described in items a) through d) shall not be counted using the method specified in this document, 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.