INTERNATIONAL STANDARD

ISO 4407

Second edition
2002-04-15

Hydraulic fluid power — Fluid contamination — Determination of particulate contamination by the counting method using an optical microscope

Transmissions hydrauliques — Pollution des fluides — Détermination de la pollution particulaire par comptage au microscope optique

Reference number
ISO 4407:2002(E)

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Printed in Switzerland

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 4407 was prepared by Technical Committee ISO/TC 131, *Fluid power systems*, Subcommittee SC 6, *Contamination control and hydraulic fluids*.

This second edition cancels and replaces the first edition (ISO 4407:1991), which has been technically revised.

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 power transmitting medium.

The presence of particulate contamination in the liquid interferes with its ability to lubricate and causes wear to the components. The level of contamination in the liquid has a direct bearing on the performance and reliability of the system, and should be controlled to a level appropriate for the system concerned.

Quantitative determination of particulate contamination requires precision in obtaining a representative sample of the liquid and in determining the level of contamination. The method of particle counting using the optical microscope is an accepted means of determining the extent of contamination. The accuracy of particle count data can be affected by the techniques used.

This International Standard details procedures for the separation of particles in liquid samples by vacuum filtration and subsequent analysis of the particles deposited on an analytical membrane filter by microscopic techniques. The techniques involve counting using transmitted or incident light both manually and using image analysis techniques. This International Standard specifies methods to ensure accurate and consistent results.

Hydraulic fluid power — Fluid contamination — Determination of particulate contamination by the counting method using an optical microscope

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulative limitations prior to use.

1 Scope

This International Standard specifies methods for determining the level of particulate contamination in liquids used in hydraulic systems by counting the number of particles deposited on the surface of a membrane filter using an optical microscope. It includes particle counting by two manual methods and image analysis, using either transmitted or incident lighting systems.

Particle sizes ⩾ 2 µm can be sized and counted by this method, but the resolution and accuracy of the results will depend upon the optical system used and the capabilities of the operator.

All hydraulic fluids with a wide range of contamination levels can be analysed according to this International Standard. However, the counting uncertainty at the larger particle sizes increases if the volume filtered is reduced to allow smaller sized particles to be counted, where a fine precipitate or a high particle concentration is present.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3722, *Hydraulic fluid power — Fluid sample containers — Qualifying and controlling cleaning methods*

ISO 4406:1999, *Hydraulic fluid power — Fluids — Method for coding the level of contamination by solid particles*

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

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

ISO 14644-1:1999, *Cleanrooms and associated controlled environments — Classification of air cleanliness*

3 Terms and definitions

For the purposes of this International Standard, the definitions given in ISO 5598 and the following apply.

3.1
blank count
count resulting from contaminants introduced from other sources, such as reagents, cleaning of glassware and preparation of the membrane filter (see 9.2)

3.2
calculation factor
ratio of the effective filtration area to the total area counted

3.3
effective filtration area
EFA
circular area of the membrane filter open to flow during filtration of liquid

NOTE Both the effective filtration area (EFA) and the effective filtration diameter (EFD) are determined in 8.2.

3.4
fibre
particle longer than 100 µm with a length-to-width ratio greater than or equal to 10:1

3.5
fixative liquid
liquid that, as a result of a heat curing process, causes a membrane filter to adhere to a glass base slide, resulting in an opaque residue

3.6
grid square
square with sides of nominally 3,1 mm printed on membrane filters

NOTE Gridded membrane filters may not be suitable for counting using image analysis techniques.

3.7
image analyser
instrumentation to automatically size and count particles deposited on a membrane filter

NOTE A video image of the particle is digitally recreated based upon the difference in the grey scale contrast of the particle and background, and the size is automatically computed. Sizing of the particle can also be undertaken on the video screen.

3.8
mountant liquid
liquid that, when heated, causes a membrane filter, previously treated with fixative liquid, to become transparent and to adhere to the cover slip (see 5.7)

3.9
particle size
size of particle as defined by the particle's longest dimension

3.10
solvent
liquid that is physically and chemically compatible with and miscible in the sample liquid

NOTE A solvent is used for diluting the sample liquid, and can be used for cleaning and rinsing the apparatus. The solvent should be chemically compatible with the apparatus, especially the membrane filter, and should not dissolve the particles.

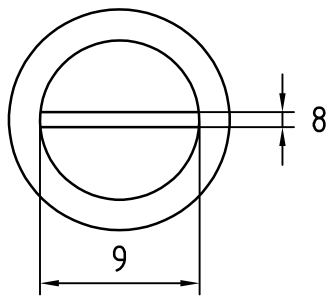
3.11
statistical counting
counting and sizing particles using a proportion of the membrane filter's surface, whereby at least 150 particles are counted over a total of at least 10 separate locations (fields)

NOTE 1 Statistical counting requires an even distribution of particles over the complete surface, and membrane filters should be rejected for counting if this is not achieved.

NOTE 2 Counting 150 particles gives a counting uncertainty of 8 %, and the counting uncertainty will be reduced if more particles are counted.

3.12
unit area
proportion of membrane filter that is counted for statistical purposes

NOTE For manual counting, the unit area is defined as the area of the membrane filter bound in the horizontal plane by two adjacent vertical membrane filter grid lines and in the vertical plane by two parallel lines either on the ocular micrometer eyepiece or drawn on a projection screen. Examples are given in Figure 1. For image analysis, this is a fixed field of view defined by the optical and electronic systems.



Key

1 Grid square
2 Width of grid square (mm)
3 Length of grid square (mm)
4 Full grid square
5 Unit area on a gridded membrane
6 Graticule height used for defining unit area (µm)
7 Unit area
8 Diametric unit area on ungridded membrane
9 Effective filtration diameter of membrane

Figure 1 — Examples of unit areas

4 Counting principle

A known volume of hydraulic liquid is vacuum filtered through a membrane filter to separate the contaminants from the liquid. The particles are examined microscopically using either incident light or transmitted light, after making the membrane filter transparent, to size and count the contaminant particles according to their largest dimension.

5 Apparatus

5.1 Drying oven, able to control temperature up to (70 ± 2) °C.

5.2 External lamp, of variable intensity, where oblique illumination of the specimen stage is required.

5.3 Filter holder, comprising:

— a funnel, 300 mL capacity with suitable calibrated volumetric graduations [e.g. (25 ± 2) mL];

— a suitable cover for the funnel (e.g. a Petri dish);

— a clamping device;

— a suitable base to support the membrane filter;

— a means of dissipating any static electricity generated during the filtering process.

5.4 Graduated cylinders, for measuring out the volume of test liquid. Either the accuracy should conform to ISO 4788 or a sample bottle calibrated with suitable volumetric graduations can be used. The accuracy of graduation should be ± 2 %.

5.5 Image analysing equipment, comprising a microscope base with a range of objective lenses and trinocular head for attaching a video camera connected to a suitable video monitor. The output of the video camera is fed to a computer which recreates a video digital image of the view, where the particles are sized and counted using specially developed software.

Although a manual X-Y stage and focus can be used, it is recommended that these be motorized and controlled by the software as this enables the particles to be localized.

5.6 Membrane filters, compatible with the sample liquid and any solvents or chemicals used in the processes. Normally, the membrane filter shall be of 47 mm diameter, white, gridded (each grid square with sides 3,08 mm ± 0,05 mm and an area equal to 1/100th of the effective filtration area), with a pore size less than 1,5 µm, used for manual counting down to 2 µm. A 47 mm diameter, white, ungridded membrane filter with a pore size less than 1,5 µm is recommended for image analysis. Membrane filters of different diameters are permissible.

It is permissible to use membrane filters of different nominal pore size to account for the minimum size of particle to be counted and the condition of the sample liquid. The pore size of the membrane filter should have a particle removal efficiency of 99,9 % at the minimum size to be counted.

The colour of the membrane filter shall be chosen for maximum contrast with the particulate contamination to be observed. For example, if most of the contaminant is translucent, transparent or white, a black membrane filter should be considered.

5.7 Microscope glass base slides and microscope glass cover slips, for transmitted-light method only, with dimensions greater than the diameter of the membrane filter. The thickness of the cover slip should be selected to ensure that the particles are in focus at the magnification used.

5.8 Membrane filter holder, plastic or equivalent with lid, for retaining the membrane filter (incident-light method only).

5.9 Microscope for particle counting, with a range of objective lenses that, in combination with the ocular lenses, are able to resolve particles $\geqslant 2$ µm. The microscope shall be equipped with:

- fine and coarse focus control;
- through-the-lens lighting for the incident light method and/or a bottom lighting source for the transmitted light method;
- a mechanical stage so that the effective filtration area of the membrane filter can be scanned;
- provision on the mechanical stage for securely holding the membrane filter holder or glass slide;
- an ocular micrometer of which the smallest division shall not subtend a distance larger than the smallest particle to be counted at a particular magnification, and with suitable graduations.

For counting with transmitted light, the optimum equipment is a projector microscope with suitable screen, over-eyepiece mirror and rotating superstage.

NOTE 1 For image analysis, it is preferable to have a stabilized lighting source controlled by the imaging software so that illumination fluctuations are eliminated and automatic correction is made for any intensity drift in the lighting source.

NOTE 2 For accurate characterization of particles using the incident light method, an additional oblique lighting source may be required (see 5.2).

Nominal magnification and optical combinations for manual counting are given in Table 1.

NOTE 3 The manufacturer of the image analysis equipment should be contacted for the magnification provided by the trinocular head video/camera combination.

Table 1 — Nominal magnifications and optical combinations

Magnification (nominal)	Ocular lens	Objective lens	Suggested minimum particle size µm
× 50	× 10	× 5	20
× 100	× 10	× 10	10
× 200	× 10	× 20	5
× 500	× 10	× 50	2

5.10 Plastic film, 0,05 mm thick × 50 mm × 50 mm, placed between the cap and neck of the sample bottle if the cap does not have an internal seal. The film shall be compatible with both the cleaning and sample liquids.

5.11 Sample bottles, 250 mL nominal capacity, preferably flat-bottomed and wide-mouthed with a screw cap containing a suitable internal polymeric seal.

5.12 Sampling agitating device, suitable for redispersing the contaminant in the liquid sample. The device, such as a laboratory bottle roller, a three-axis paint shaker or an ultrasonic bath rated at 3 000 W/m^2 to 10 000 W/m^2 of base area, shall not alter the basic size distribution of the contaminant.

5.13 Solvent dispenser, a pressurized vessel that discharges solvent (see 6.1.4) through an in-line membrane filter with a pore size not greater than 1 µm.

5.14 Stage micrometer, graduated in 0,1 mm and 0,01 mm divisions, calibrated and traceable to national standards.