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

Transmissions hydrauliques — Pollution des fluides — Détermination de la pollution particulaire solide — Méthode de comptage au microscope

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Foreword

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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.

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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.

Reliable system performance requires control of the fluid medium. Qualitative and quantitative determination of particulate contamination in the fluid medium requires precision in obtaining the sample and determining the nature and extent of contamination.

The microscope method of determining fluid contamination involves counting illuminated filtered particles under a microscope. The method employs membrane filters, which maintain fluid cleanliness by removing insoluble particles.

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

1 Scope

This International Standard defines methods of determining the solid particle contamination of hydraulic fluids used in hydraulic fluid power systems by counting the number of particles on the surface of a membrane filter using a microscope under transmitted light and under incident light.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4021:1977, *Hydraulic fluid power — Particulate contamination analysis — Extraction of fluid samples from lines of an operating system.*

ISO 5598:1985, *Fluid power systems and components — Vocabulary.*

3 Definitions

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

3.1 blank count: Count resulting from contaminants introduced from other sources, such as reagents or cleaning of glassware, which do not exceed 1 000 particles, the size of which are 5 µm or greater.

NOTE 1 For this determination, a clean, unused membrane is prepared as indicated in clause 9, replacing the sample fluid by the same volume of filtered solvent. Counts in excess of 1 000 particles indicate unsatisfactory test conditions; the required standard should be achieved before sample analysis is undertaken.

3.2 calculation factor: Ratio of the effective area to the total area counted.

3.3 effective area: Area of the membrane open to flow during filtration of the fluid. (This is generally accepted as 100 grid squares and has an area of 890 mm².)

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

3.5 fixative fluid: Fluid which, as a result of a heat-curing process, causes a contaminated membrane filter to adhere to a glass base slide, resulting in an opaque honeycomb residue.

3.6 grid square: Square that has sides of 3,08 mm and is printed on the membrane filter.

3.7 mountant fluid: Fluid which is applied to the dry honeycomb residue on the glass base slide to render it transparent.

3.8 particle size: Size of particle determined by measurement using a calibrated ocular micrometer.

3.9 sub-unit area: Area approximately one-twentieth of a grid square used where populations of 25 000 and above are estimated at ×10 magnification. (See 10.2.)

3.10 unit area: Area bounded in the horizontal plane by two adjacent vertical membrane grid lines and in the vertical plane by two parallel lines in the circular micrometer eyepiece or drawn on the projection screen. The dimensions of the unit area are measured by a previously calibrated ocular micrometer.

4 Counting principle

Filtration of a known volume of hydraulic fluid through a membrane filter under vacuum conditions to collect contaminants on a filter surface. Examination of the membrane microscopically by incident light or, having made it transparent, by transmitted light, to measure, count and size the contaminant particles according to their largest dimension.

5 Apparatus

5.1 Filter holder, comprising

— a graduated glass measuring funnel, of 250 ml capacity;

— a clamping device;

— a suitable base to support the membrane.

5.2 Cap for the funnel

5.3 Filter membrane, 47 mm in diameter, white, gridded, with less than 1 µm pore diameter, each grid square having sides of 3,08 mm and equal to one-hundredth of the effective area of the membrane.

NOTE 2 If the selected method of rendering the membrane transparent implied chemical filter destruction, the membrane filter should have the same characteristics but should not be gridded.

5.4 Vacuum flask, 1 litre capacity.

5.5 Device, capable of establishing a vacuum of 86,6 kPa (0,866 bar)[1] (i.e. 650 mmHg).

5.6 Filtered-solvent dispenser, (i.e. a pressure-operated system which discharges the solvent through a filter membrane, the pore diameter of which is less than or equal to 1,2 µm).

5.7 Flat-ended tweezers, of stainless steel.

5.8 Microscope glass base slides and **microscope glass cover slips**, thickness 0,25 mm, (for transmitted-light method only).

5.9 Petri dishes, plastic or equivalent, for 47 mm membrane filters (for incident-light method only).

1) 1 bar = 10^5 Pa

5.10 Drying oven, with temperature control up to 70 °C.

5.11 Microscope, with ocular micrometer eyepiece capable of resolving particles down to 5 µm, fitted with a mechanical stage so that the effective area can be scanned. The nominal magnifications and optical combinations given in table 1 or table 2 are recommended. The optimum equipment is a projector microscope with suitable screen, over-eyepiece mirror and rotating superstage.

Table 1 — Nominal magnifications and optical combinations for transmitted-light method

Magnification		Ocular	Objective
nom.	tol.		
×100	± 10	×10	×10
×200	± 20	×10	×20
×450	± 50	×10	×45

Table 2 — Nominal magnifications and optical combinations for incident-light method

Magnification		Ocular	Objective
nom.	tol.		
×50	± 10	×10	×5 ± 1
×100	± 10	×10	×10 ± 1
×200	± 20	×10	×20 ± 2

5.12 Stage micrometer, graduated in 0,1 mm and 0,01 mm divisions.

5.13 External lamp, of variable intensity, if the microscope does not include any light source through the lenses (for incident-light method only).

5.14 Lamp, suitable to be used as a light source (for transmitted-light method only).

5.15 Tally counter

5.16 Sample bottles, recommended capacity 250 ml, preferably wide-mouthed with a screw cap.

5.17 Sample bottles, at least 100 ml capacity, permanently marked to indicate 100 ml sample size, with screw caps.

5.18 Plastic films, 0,05 mm thick × 50 mm × 50 mm, to be placed between the cap and the neck of the sample bottles.

6 Reagents

6.1 Rinsing and cleaning chemicals

6.1.1 Liquid detergent, without solid residue.

6.1.2 Distilled or **demineralized water.**

6.1.3 Isopropyl alcohol, acetone free.

6.1.4 Petroleum ether (boiling point 100 °C to 120 °C) or **trichlorotrifluoroethane** (F.113) .

WARNING — Exercise care when using solvents which have low flash points. Appropriate precautions should also be taken to avoid inhalation of toxic fumes emanating from these solvents.

6.2 Fluids for the sampling procedure (for the transmitted-light method)

6.2.1 Fixative fluid (see 3.5).

6.2.2 Mountant fluid, having a refractive index similar to that of the glass cover slip (see 3.7).

7 Glassware cleaning procedure

Clean the filtration apparatus (5.1), bottles (5.16), glass slides and cover slips (5.8) and Petri dishes (5.9) as follows:

a) wash the glassware in warm tap water/liquid detergent (6.1.1) solution;

b) rinse three times with distilled or demineralized water (6.1.2);

c) rinse three times with filtered isopropyl alcohol (6.1.3) to remove water;

d) rinse three times with filtered trichlorotrifluoroethane or petroleum ether (6.1.4) (check that petroleum ether does not leave greasy traces), and

— for the filtration apparatus, turn the funnel upside down for 15 s to allow the solvent to drain and evaporate;

— for the bottles, leave a little solvent in the bottle and cover by inserting a plastic film (5.18) (rinsed with the filtered solvent) between the neck and stopper.

NOTE 3 Solvent evaporation slightly pressurizes the bottle and therefore precludes contamination when the bottle is opened.

8 Sampling

Extract the sample fluid in accordance with the method laid down in ISO 4021.

9 Procedure

Carry out the following test procedure in a clean room or cabinet.

9.1 Preparation

9.1.1 Using clean tweezers (5.7), remove a membrane filter (5.3) from its container and place it centrally on the screen of the funnel support assembly (5.1). Carefully install and clamp the funnel in position. Do not remove the cap (5.2) from the funnel opening until ready to start filtration.

9.1.2 After noting any details, remove any tied-up or loosely attached labels, ensuring that sample identity is retained.

9.1.3 Thoroughly clean the outside of the sample bottle in the region of the cap by washing with filtered petroleum ether or approved alternative. Shake the bottle vigorously for at least 1 min. Remove cap and film, if fitted, and rinse off any matter adhering to the bottle neck threads with a jet of filtered petroleum ether or approved solvent, ensuring that none of the washings enters the bottle.

9.1.4 Connect the vacuum device (5.5) to the side arm of the vacuum flask (5.4). Pour 100 ml from the sample bottle into the filter funnel. Apply vacuum. When the funnel contents have been reduced to a small volume, rinse down the funnel walls with a spirally directed jet of filtered solvent, taking care not to disturb the particulate matter on the membrane filter. Aspirate to dryness and prepare the membranes following the chosen method of observation (9.2 or 9.3).

9.2 Observation under transmitted light

9.2.1 Take a glass base slide and apply sufficient fixative fluid (6.2.1) onto it.

9.2.2 Using the tweezers, carefully remove the contaminated membrane filter and place it with the contaminated surface uppermost onto the fixative fluid on the glass base slide, aligning the grid on the membrane filter parallel with the edge of the slide.

9.2.3 Place the glass base slide carrying the contaminated membrane filter, suitably covered, in the drying oven (5.10) regulated to a temperature of 55 °C to 60 °C and allow to dry for approximately 1 h. The drying time is not critical, but when the contaminated membrane filter has become fixed to the glass slide, it should be opaque and white in colour.

9.2.4 When dry, remove the base slide from the oven and allow to cool for 2 min to 3 min. During this cooling period, take a glass cover slip and apply sufficient mountant fluid (6.2.2) onto it.

9.2.5 Take the slide onto which the membrane has been bonded and lower the side of the cover slip carrying the mountant onto the membrane and allow to settle with the sides of both glasses in alignment.

NOTE 4 Care should be taken to avoid crushing particles under the cover slip.

9.2.6 Carefully return the assembled microscope slides to the oven, regulated to a temperature of 55 °C to 60 °C, for at least 90 min. A longer period of time (up to 36 h) is desirable and will improve the permanency of the mount.

9.2.7 When the drying period has been completed, remove the assembled microscope slides from the oven and allow to cool.

9.2.8 Register and mark the assembled microscope slides for identification.

9.3 Observation under incident light

9.3.1 Using tweezers with unserrated tips, carefully place the membrane onto a clean Petri dish.

9.3.2 Register and mark the slide for identification.

9.4 Blank tests

9.4.1 Using the tweezers, remove one membrane filter from its container. Rinse the top surfaces with filtered solvent (if a solvent spray gun is used, use a nozzle giving a conical jet so as not to break the filter). Place the membrane filter on the filter-holder base with the printed grid side up.

9.4.2 Lower the funnel into the filter without sliding it and secure with the clamping device.

9.4.3 Pour a volume of filtered solvent identical to the fluid sample volume into a clean sample bottle and shake it. Remove the funnel cap and pour the contents of the bottle into the funnel. Pour approximately 50 ml of the filtered solvent into the bottle. Shake the solvent and pour it into the funnel. Replace the funnel cap.

9.4.4 Apply a vacuum and filter the solution until about 50 ml is left in the funnel. Turn off the vacuum. Remove the funnel cap, rinse the funnel walls with the filtered solvent and replace the funnel cap.

9.4.5 Apply a vacuum and filter the solution until complete fluid filtration. Wait until the filter is completely dry. Turn off the vacuum. Remove the funnel cap, the clamp device and the funnel.

9.4.6 Proceed as described in 9.2 or 9.3.

10 Microscope calibration

10.1 Using the stage micrometer (5.12), calibrate the microscope (5.11) for sizing particles at the following magnifications: ×50, ×100, ×200 and ×450.

10.2 For each magnification, measure the width of unit and sub-unit areas which are less than a grid square. If a screen is used, mount it perpendicular to the direction of projection and at a distance to give the enlargement required. Horizontal lines can then be inscribed on this screen to give the smaller area required. If a microscope is fitted with a second micrometer filar eyepiece, this may be used to obtain the required areas.

11 Particle counting and sizing procedure

11.1 Particle size ranges

Classify the particles in the following size ranges:

5 µm < particle size ≤ 15 µm
15 µm < particle size ≤ 25 µm
25 µm < particle size ≤ 50 µm
50 µm < particle size ≤ 100 µm
particle size > 100 µm
fibres (see 3.4)

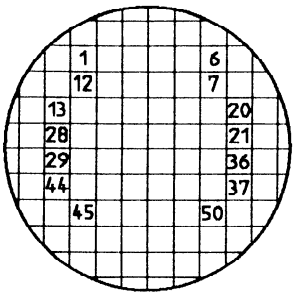
11.2 Selection of nominal magnification

Select from table 3 the magnification appropriate to the size range to be counted.

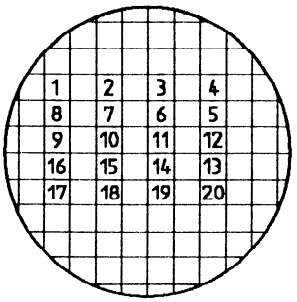
11.3 Counting by unit area

11.3.1 Check the entire membrane for good particle distribution. If homogeneous, count as in 11.3.2. If not, count the whole membrane or prepare another sample.

11.3.2 Scan the first unit area and count the number of particles using the sizing criteria as specified in 3.9. A typical counting pattern is shown in figure 1. Continue counting unit areas. If in the first 10 unit areas, the total number of particles exceeds



50 SQUARES



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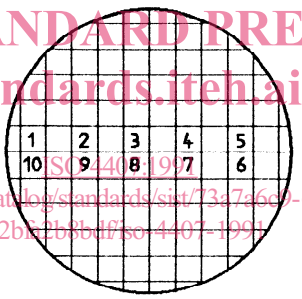

10 SQUARES

Figure 1 — Typical counting patterns for grid squares

Table 3 — Particle size ranges and nominal magnifications

Size range µm	Nominal magnification	
	Transmitted light	Incident light
5 < particle size ⩽ 15[1)]	×450	×200
15 < particle size ⩽ 25	×200	×100
25 < particle size ⩽ 50	×200	×100
50 < particle size ⩽ 100	×100	×50
particle size > 100	×100	×50
fibres	×100	×50

1) Where required, this range may be sub-divided into 5 µm < particle size ⩽ 10 µm and 10 µm < particle size ⩽ 15 µm size ranges.

300 in a given size range, the count is considered complete for that size range. If this number is not reached, further well-scattered unit areas are to be counted until either:

a) a total of 300 particles has been exceeded, or

b) 100 unit areas have been counted.

NOTE 5 If the number of particles per unit area exceeds 50, the count can be used on sub-unit areas.

11.4 Calculation of total count

Obtain total counts by multiplying the obtained count by the calculation factor F_c.

$$F_c = \frac{890\ \text{mm}^2}{l \times w \times n}$$

where