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# Hydraulic fluid power — Fluid contamination — Determination of particulate contamination by the gravimetric method

*Transmissions hydrauliques — Pollution des fluides — Détermination de la pollution particulaire par la méthode gravimétrique*

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# Contents

# Foreword

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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).

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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 second edition cancels and replaces the first edition (ISO 4405:1991), which has been technically revised.

The main changes are as follows.

- The calibration and test equipment have been updated and reduced to the most essential parts.
- The single steps of the calibration and test procedure have been updated and adapted to achieve the most reliable and verifiable test results.
- The double-membrane method has been eliminated as the test results have proven to be less reliable than those obtained by the single-membrane method.

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.

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 in determining the nature and extent of contamination.

The gravimetric method of determination of fluid contamination involves weighing suspended solids per unit volume of fluid. The method employs membrane filters, which maintain fluid cleanliness by removing insoluble particles.

# Hydraulic fluid power — Fluid contamination — Determination of particulate contamination by the gravimetric method

## 1 Scope

This document defines the gravimetric method for determining the contamination level of fluids used in hydraulic fluid power systems.

The working instructions provided in this document serve for the gravimetric determination of dirt content of pressure fluids from mineral oil with additives. They are used in hydraulic systems with hydrostatic drive.

## 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 4021, *Hydraulic fluid power — Particulate contamination analysis — Extraction of fluid samples from lines of an operating system*

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

ISO 11158, *Lubricants, industrial oils and related products (class L) — Family H (hydraulic systems) — Specifications for categories HH, HL, HM, HV and HG*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5598 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**
**hydraulic oil HH**
non-inhibited mineral oil

**3.2**
**hydraulic oil HL**
oil of HH type with improved anti-rust and anti-oxidation properties

**3.3**
**hydraulic oil HM**
oil of HL type with improved anti-wear and filterability properties

**3.4**
**hydraulic oil HV**
oil of HM type with improved viscosity/temperature properties

**3.5**
**hydraulic oil HG**
oil of HM type with improved anti-stick-slip properties

# 4 Principle

The gravimetric amount of contamination shall be determined by filtering a defined volume of fluid under vacuum conditions through one filter membrane. The increase in mass of the membrane after filtration represents the solid impurity content. This analytical method is suitable for dirt concentrations of 3 mg/l or greater and the hydraulic oils of categories: HH, HL, HM, HV, and HG, in accordance with ISO 11158. The room conditions should have a temperature between 20 °C and 30 °C and humidity between 30 % and 65 % relative humidity (RH).

# 5 Test apparatus

## 5.1 Equipment

1) One suction bottle made of glass.
2) Solvent dispenser rinsed and filled with filtered solvent - 0,2 µm (see Figure C.1).
3) PTFE seal (white) for stainless steel filter grid or glass frit (see Figure C.2).
4) Membrane holder funnel made of glass for the stainless steel filter grid (see Figure C.3).
5) Stainless steel filter grid or glass frit (see Figure C.4 or Figure C.5).
6) Glass funnel.
7) Metal spring clip with electrical grounding and glass funnel (see Figure C.6).
8) Petri dish made of glass with lid (see Figure C.7).
9) Watch glass made of glass; diameter approximately 60 mm (see Figure C.8).
10) Exsiccator/desiccator with silica gel drying agent (with colour indicator), to operate without vacuum (see Figure C.9).
11) One 250 ml graduated cylinder for oil samples (see Figure C.10).
12) Vacuum pump with adjustable vacuum level.
13) Non-ventilated drying oven up to 100 °C.
14) Filter tweezers, stainless steel with smooth endings.
15) Solvent-proof gloves without powder inside.
16) Analytical balance (accuracy ±0,02 mg with five-digit display), the electrostatic conductivity between the weighting plate and the grounding of the power plug shall be given.
17) Clock accurate to the second.

For photos and description of the test equipment, see Annex C.

## 5.2 Filter membrane

Filter membranes, of 47 mm diameter, white, non-gridded 0,8 µm pore size and compatible with the fluid to be analysed and with the rinsing chemicals can be used. Reference membranes have a 0,8 µm pore size. The material shall be made of cellulose acetate, for example, cellulose mixed ester

0,8 µm from Millipore (Type AAWP04700), Pall (Type GN4-08 µm Metricel MCE) or Sartorius (item name: Zelluloseacetat-Filter 0,8 µm) and for the gravimetric determination, the use of the membrane AAWP04700 from Millipore[1].

## 6 Rinsing and cleaning chemicals (Solvent)

The solvent to use is Petrol 40/65, petroleum ether 40/65 or 40/60 filtered through a 0,2 µm membrane filter. No other solvent is acceptable unless it is qualified in Annex A.

NOTE It is useful to take hexane-free petrol, hexane is harmful to health.

**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.**

## 7 Glassware cleaning procedure [determination of the cleanliness of the glass funnel and the graduated cylinder at the beginning (blank value)]

Before the operation, all the glassware shall be cleaned with filtered solvent. To determine the cleanliness, i.e. the blank value of the glass funnel and the graduated cylinder by the gravimetric analysis, proceed as follows.

1) Put one membrane onto the glass membrane holder funnel with the stainless steel filter grid or the glass frit. Apply vacuum and rinse (20-30) ml with solvent.

2) Remove the membrane and lay it on the watch glass. Lay the watch glass with the membrane in a suitable Petri dish and dry in the oven (half covered) at approximately 80 °C for a minimum of 10 min.

3) Subsequently put the watch glass with the membrane into the exsiccator/desiccator and let it cool down for a minimum of 10 min.

4) Take the watch glass out of the exsiccator/desiccator and lay the watch glass with the membrane in a suitable (half covered) Petri dish condition for approximately 10 min at room temperature. Weigh without watch glass. Record displayed value when value no longer changes.

5) Place the conditioned membrane on the glass membrane holder funnel with the stainless steel filter grid or glass frit. Attach the glass funnel.

6) Rinse the graduated cylinder with solvent. Transfer the rinsing volume into the filtration unit. Apply vacuum and begin the filtration. Reduce the vacuum to achieve a rinsing time of (50-100) ml solvent of at least 4 min. Rinse three times with approximately 10 % of the graduated cylinder volume. This procedure ensures that the oil is dissolved out of the membrane without leaving any residue.

7) Remove the glass funnel. Let the liquid drop onto the membrane, rinse carefully the edge of the filter membrane with fluid out of the solvent dispenser.

8) Place the membrane onto a labelled watch glass. Place the watch glass with the membrane in a suitable Petri dish and dry in the oven (half covered) at approximately 80 °C for a minimum of 10 min.

9) Subsequently, put the watch glass with the membrane into the exsiccator/desiccator and let them cool down for a minimum of 10 min.

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[1] These are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products named. Equivalent products may be used if they can be shown to lead to the same results.

10) Take the watch glass out of the exsiccator/desiccator, condition at room temperature for approximately 10 min. Weigh the membrane without the watch glass. Record the displayed value when the value no longer changes. The performance analysis shall be carried out according to Table B.1.

If the blank value is higher than the permitted 0,1 mg, the glasses shall be cleaned and the solvent is to be filtered anew, if necessary. Subsequently, the blank value is to be determined again.

## 8 Sampling

**8.1** Ensure that the samples are as representative as possible of the fluid under consideration.

Ensure that the sampling procedure to be devised by each society or laboratory allows good repeatability.

Check the sampling procedure periodically by collecting two samples and making two different measurements on the same sample.

**8.2** Extract 500 ml of fluid from an operating hydraulic fluid power system according to the method described in ISO 4021. This volume may, however, be modified to suit very different contamination levels.

In all cases, the sample volume used for the measurement shall be stated with a tolerance of ±1 %.

## 9 Conditioning and weighing the analysis membrane

Prior to the test the membranes used shall be conditioned to ensure representative and reproducible results. To do so, proceed as follows.

Move the membrane with the filter tweezers only. Each membrane shall be laid onto a separate, labelled watch glass. Touch the watch glass with gloves only.

1) Label the watch glass.
2) Do not condition more than four membranes at once.
3) Place one membrane onto the filter grid or glass frit, apply the vacuum and rinse with approximately 50 ml of solvent.
4) Remove the membrane and lay it onto the watch glass. Lay the watch glass with the membrane in a suitable Petri dish (half covered) and dry in the oven at approximately 80 °C for a minimum of 10 min.
5) Put the watch glass with the membrane into the exsiccator/desiccator and let them cool down for a minimum of 10 min.
6) Take the watch glass out of the exsiccator/desiccator and lay the watch glass with the membrane in a suitable (half covered) Petri dish, condition at room temperature for approximately 10 min. Weigh the membrane without the watch glass. Record the displayed value when the value does not change anymore.

## 10 Blank test

It can be helpful to perform a blank test to be sure that the test procedure works. Proceed exactly as described in Clause 11. Do not use oil, only the solvent. The blank test is passed if the analysed value is equal or less than 0,1 mg.

## 11 Sample analysis

1) Shake the sample for 2 min in all directions.

2) Pour (200-250) ml into the graduated cylinder. Let the sample degas (visual test). Determine the sample volume from the graduated cylinder scale and record it using Table B.2.

   NOTE If an increased dirt concentration of the sample is recognizable, then it is possible to work with a smaller sample quantity.

3) Place the conditioned membrane (see Clause 9) on the glass membrane holder funnel with the stainless steel filter grid or glass frit. Attach the glass funnel.

4) Pour the whole sample volume of the graduated cylinder into the glass funnel, apply the vacuum and start the filtration.

5) After the whole filtration of the sample volume, rinse the graduated cylinder carefully with solvent. Rinse three times with approximately 10 % of the graduated cylinder volume.

6) Rinse the inner surface of the glass funnel with approximately 50 ml of solvent. Do not remove the glass funnel.

7) Add 200 ml of solvent to the glass funnel. Attention: reduce the vacuum level of the vacuum pump to achieve a rinsing time of the 200 ml of solvent of at least 4 min. (The conditions required to achieve a rinsing time of at least 4 min for 200 ml of solvent should be locally established by the laboratory).

8) After the whole filtration of 200 ml of solvent rinse again the glass funnel with approximately 50 ml of solvent. Remove the glass funnel and increase the vacuum on the vacuum pump.

   Attention: carefully also rinse the bottom edge of the funnel with solvent. Let the liquid drop onto the membrane.

9) Carefully rinse the edges of the membranes with solvent from out to in to remove all the oil (approximately 20 ml or more). Ensure that no dust is rinsed from the membrane.

10) Place the membrane onto the labelled watch glass. Place the watch glass with the membrane in a suitable Petri dish and dry in the oven (half covered) at approximately 80 °C for a minimum of 10 min.

11) Subsequently put the watch glass with the membrane into the exsiccator/desiccator and let them cool down for a minimum of 10 min.

12) Take the watch glass out of the exsiccator/desiccator and lay the watch glass with the membrane in a suitable (half covered) Petri dish, condition at room temperature for approximately 10 min. Weigh the membrane without the watch glass. Record the displayed value when the value does not change anymore. The performance analysis shall be carried out according to Table B.2.

13) Safely dispose of the mixture solvent and oil in accordance with local hazardous material procedures.

## 12 Test result

**12.1** The solid impurity content of the sample, expressed in mg per 1 000 ml of fluid, is given by the formulae presented in Annex B.

## 13 Test repeatability

In order to obtain repeatable and representative test results, it is necessary that the tests are performed on the same day and without interruption. Compare two measurements on the same sample made by the same operator. If they differ by more than a mass fraction of 5 %, repeat the operation.

## 14 Identification statement (reference to this document)

Use the following statement in test reports, catalogues and sales literature when electing to conform to this document:

“Determination of hydraulic fluid contamination by the gravimetric method performed in accordance with ISO 4405, *Hydraulic fluid power — Fluid contamination — Determination of particulate contaminants by the gravimetric method”*.

# Annex A
(normative)

# Use of different solvent

## A.1 General

This procedure for the qualification of alternative solvents applies when the standardized solvents listed in Clause 6 cannot be used as solvent.

## A.2 Test procedure

The gravimetric content of contamination in the oil sample shall be determined with the standardized method and solvent petrol (see Clause 6) described in this document with an ISO MTD (ISO 12103-1–A3) concentration of 3 mg/l.

Select a different solvent that could be a substitute. The analysis according to the standardized method, starting with Clause 7, shall also be conducted with this alternative solvent using the same gravimetric concentration range of the oil sample and same concentration of ISO MTD as for the standardized solvent.

The tests shall be conducted by the same operator.

## A.3 Data analysis and acceptance criteria

There shall be at least three results with the alternative solvent from one oil sample. The acceptance criteria is based upon the 95 % confidence interval using the statistical t-test.

Compare the test results of the standardized and substitute solvents. The alternative solvent is accepted when the average of the individual results of this analysis, compared to the average of the individual results of the standardized solvent, it should fit in 95 % confidence interval using two sample t-test.

When the conditions are fulfilled for an oil sample with a concentration of 3 mg/l, this solvent may also be used for oil samples with higher concentrations.

# Annex B
## (normative)

# Data recording

General information:

- Laboratory:
- Date:
- Solvent:
- Humidity (%):
- Temperature (°C):

**Table B.1 — Determination of the blank value**

| | **Weight membrane after conditioning,** $m_{BV(2)}$ (mg) | **Weight membrane after filtration,** $m_{BV(1)}$ (mg) | **Difference** ($m_{BV(1)} - m_{BV(2)}$) (mg) |
|---|---|---|---|
| Blank value (BV) | | | |
| Remark | | | |

Limit value BV: 0,1 mg

**Table B.2 — Performance analysis, mg/l**

| **Sample no.** | **Sample volume,** $V$ (ml) | **Membrane no.** | **Weight membrane after conditioning,** $m_{OM(2)}$ (mg) | **Weight membrane, after filtration,** $m_{OM(1)}$ (mg) | **Total weight,** $m_{ges}$ (mg)[a] | **Result,** $c$ (mg/l)[b] |
|---|---|---|---|---|---|---|
| 1,1 | | | | | | |
| 1,2 | | | | | | |
| Average | | | | | | |
| Remark | | | | | | |

[a] Calculation test result: $m_{ges} = m_{OM(1)} - m_{OM(2)}$

[b] Calculation test result: $c = \frac{m_{ges}}{V} \cdot 1\,000$

# Annex C
(informative)

## Photographs of the test equipment

Figures C.1 to C.10 provide photographs of the test equipment described in Clause 5.

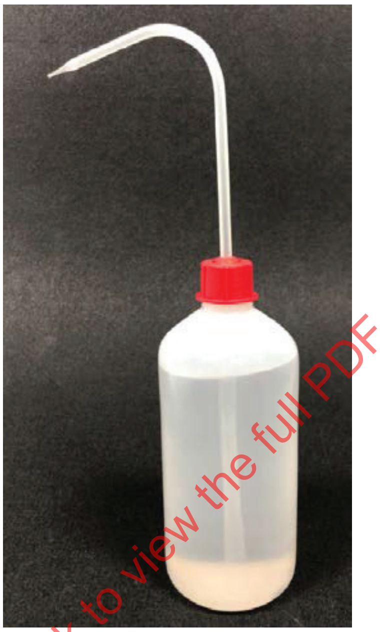

**Figure C.1 — Solvent dispenser, rinsed with filtered solvent (0,2 µm)**

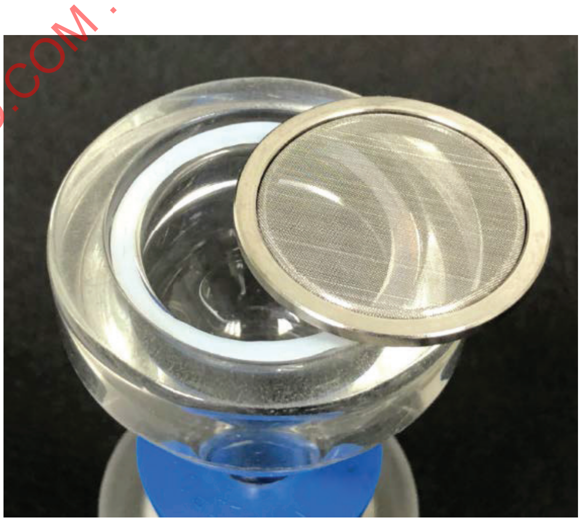

**Figure C.2 — PTFE seal (white) for stainless steel filter grid (see Figure C.4)**

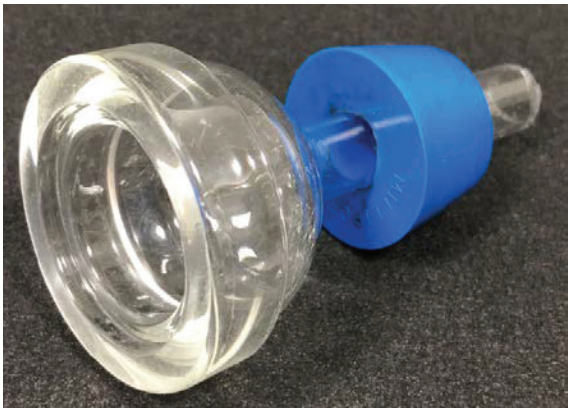

Figure C.3 — Membrane holder funnel made of glass for the stainless steel filter grid

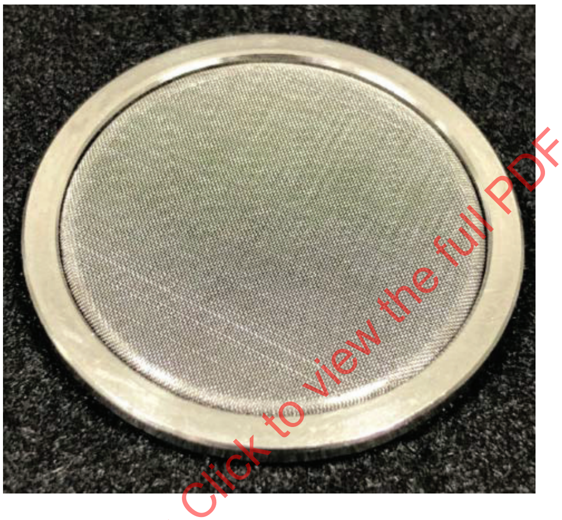

Figure C.4 — Stainless steel filter grid

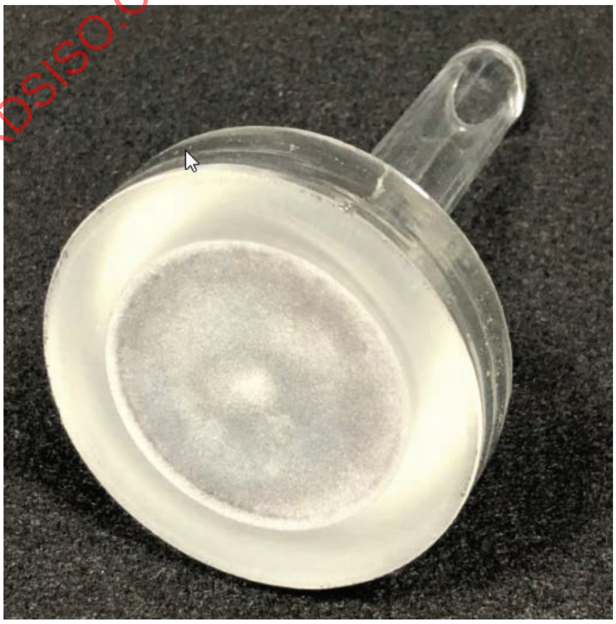

Figure C.5 — Membrane holder funnel: glass frit