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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

ICS: 23.100.60

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

The main changes compared to the previous edition are as follows:

— The calibration and test equipment were updated and reduced to the most essential parts

— The single steps of the calibration and test procedure were updated and adapted to achieve the best reliable and validatable test results.

— The double-membrane method was eliminated as the test results proved to be not that reliable as 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 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 International Standard defines the gravimetric method for determining the contamination level of fluids used in hydraulic fluid power systems.

This working instruction serves 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 6743-4, *Lubricants, industrial oils and related products (class L) — Classification — Part 4: Family H (Hydraulic systems)*

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 terminological 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

Perform filtration of a known 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. The room conditions should have a temperature between 20 and 30°C and humidity between 30 and 65 % RH.

5 Test Apparatus

5.1 Equipment

1. One litre suction bottles out of glass
2. Solvent dispenser (see Figure C.1), rinsed with filtered petrol (0,2 µm)
3. PTFE seal (white) for stainless steel filter grid or glass frit (see Figure C.2)
4. Membrane holder funnel out of glass (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 (see Figure C.6)
8. Petri dish out of glass with lid (see Figure C.7)
9. Watch glass out 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 (see Figure C.10) for oil samples
12. Vacuum pump with adjustable vacuum
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 must 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 47mm 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 must be out of cellulose acetate. [1)]

6 Rinsing and cleaning chemicals (Solvent)

The solvent to use is Petrol 40/65, petroleum ether 40/65 or 40/60 (it is useful to take hexane-free petrol, hexane is harmful to health) filtered through a 0,2µm membrane filter. No other solvent is acceptable unless it is qualified in Annex A.

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 operation all the glass ware is to 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 minimum of 10 min.

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

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 does not change any more.

5) Place the conditioned membrane on the glass membrane holder funnel with the stainless steel filter grid or glass frit. Attach 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 get 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 10 min as the minimum.

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

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1) For example cellulose mixed ester 0,8 µm from Millipore (Type AAWP04700), Pall (Type GN4-08µm Metricel MCE) or Sartorius (item name: Zelluloseacetat-Filter 0,8 µm) and for the gravimetric determination the use of the membrane AAWP04700 from Millipore. 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 displayed value when the value does not change any more. The performance analysis will be carried out according to Table B.1.

If the blank value is higher than the permitted 0,1 mg, the glasses are to 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 by the method described in ISO 4021.

NOTE 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 need to be conditioned to ensure representative and reproducible results. To do so, proceed as follows:

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

1) Label watch glass.

2) Do not condition more than 4 membranes at once!

3) Place one membrane onto the filter grid or glass frit, apply vacuum and rinse with approximately 50 ml of solvent.

4) Remove the membrane and lay them 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 10 min as the minimum.

5) Put the watch glass with the membrane into the exsiccator/desiccator and let them cool down for 10 min as the minimum.

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 any more.

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 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). Read and record the sample volume in, see 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 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 of the vacuum pump to get a rinsing time of the 200 ml of solvent of at least 4 min. (The conditions required to get a rinsing time of at least 4 min for 200 ml of solvent should be locally established by the performing activity or 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 10 min as the minimum.

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

12) Take the watch glass out of the exsiccator/desiccator and lay the watch glass with the membrane in a suitable (half covered) Peri 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 any more. The performance analysis will be carried out according to Table B.2.

13) Dispose of the mixture of solvent and oil.

NOTE 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 milligrams per 1000 ml of fluid, is given by the formulae, presented in Table B.2.

13 Test repeatability

In order to obtain reproducible 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 5 % (*m/m*), repeat the operation.

14 Identification statement (Reference to this International Standard)

Use the following statement in test reports, catalogues and sales literature when electing to comply with 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

Determine the gravimetric content of dirt in your oil sample with the standardised method (ISO 4405). Select a different solvent that you think could be a substitute. With this solvent, perform the analysis according to the standardised method (start with Clause 7).

There should be at least three results with this different solvent from one oil sample. If they differ by more than 5 % (*m/m*) repeat the operation. Compare the two solvents made by the same operator. The different solvent is accepted if the average of the individual results of this analysis, compared to the average of the individual results of the standardized solvent is less than 5 %.

The different solvent is only permitted if the comparative analysis of the solvents was performed in the same gravimetric concentration range of the oil sample. If an oil sample with a concentration of 3 mg/l was selected for the comparative analysis of the solvents and the above-mentioned conditions were fulfilled, this solvent is also suitable 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 [mg] (m_{BV2})	Weight membrane, after filtration [mg] (m_{BV1})	Difference [mg] ($m_{BV1} - m_{BV2}$)
Blank value (BV)			
Remark:			

Limit value BV: 0,1mg

Table B.2 — Performance analysis, mg/l

Sample no.	Sample volume [ml] (V)	Membrane no.	Weight membrane, after conditioning [mg] (m_{OM2})	Weight membrane, after filtration [mg] (m_{OM1})	Total weight [mg] (m_{ges})*	Result [mg/l] (c)*
1.1						
1.2						
Remark:						

*Calculation test result:

$$\left(m_{OM_1} - m_{OM_2}\right) = m_{ges}$$

$$c[\mathrm{mg/l}] = \left(m_{ges}[\mathrm{mg}] / V[\mathrm{ml}]\right) \cdot 1000 [\mathrm{ml/l}]$$

Annex C
(informative)

Photos of the test equipment

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

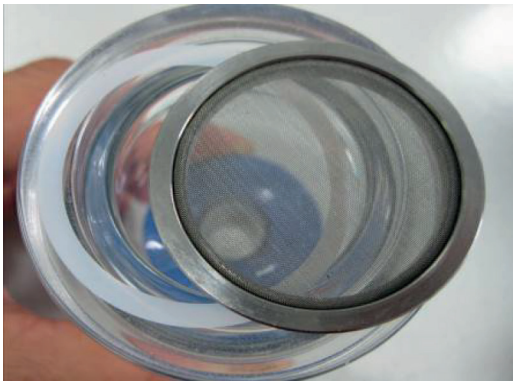

Figure C.2 — PTFE seal (white) for stainless steel filter grid (see C.4)

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

Figure C.4 — Stainless steel filter grid

Figure C.5 — Membrane holder funnel: glass frit

Figure C.6 — Metal spring clip with electrical grounding and glass funnel

Figure C.7 — Petri dish out of glass with lid

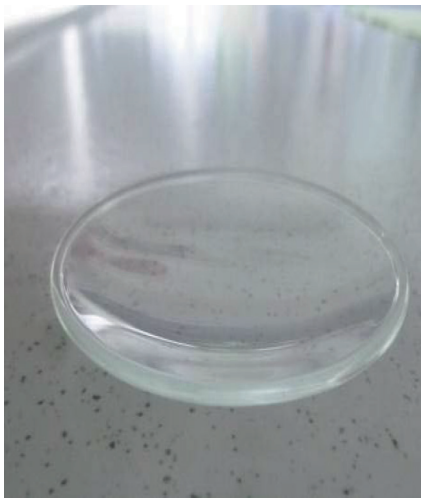

Figure C.8 — Watch glass out of glass diameter approximately 60 mm

Figure C.9 — Exsiccator/desiccator

Figure C.10 — Graduated cylinder