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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*



Reference number
ISO 4405:1991(E)

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.

International Standard ISO 4405 was prepared by Technical Committee ISO/TC 131, *Fluid power systems*.

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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 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.

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

1 Scope

This International Standard defines two gravimetric methods for determining the contamination level of fluids used in hydraulic fluid power systems.

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 3938:1986, *Hydraulic fluid power — Contamination analysis — Method for reporting analysis data*.

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 apply.

4 Principle

Filtration of a known volume of fluid under vacuum conditions through one or two identical superimposed filter membranes. The increase in mass of the

membrane or the difference in mass of the two membranes after filtration represents the solid impurity content.

5 Apparatus

5.1 Filter holder, comprising

- a graduated glass funnel, of 250 ml capacity;
- a clamping device;
- a glass base including a sintered glass or stainless steel filter-holder grid.

5.2 Cap for the funnel, for example, the lid of a Petri dish.

5.3 Filter membranes, of 47 mm diameter, white, non-gridded and compatible with the fluid to be analysed and with the rinsing chemicals. Reference membranes have a 0,8 µm pore size. Any other pore size used shall be stated.

5.4 Filter flask.

5.5 Device, for establishing a vacuum of 86,6 kPa (0,866 bar¹⁾) (i.e. 650 mmHg).

5.6 Filtered-solvent dispenser, (i.e. a pressure-operated system which discharges the solvent through a filter membrane).

5.7 Flat-ended tweezers, of stainless steel.

5.8 Petri dishes, glass, 150 mm in diameter.

5.9 Glass flasks, narrow-necked and with screw caps, at least 100 ml in capacity, with an indelible gauge mark at the level of 100 ml.

1) 1 bar = 10⁵ Pa.

5.10 Plastic films, 0,05 mm thick $\times$ 50 mm $\times$ 50 mm, to be placed between the cap and the neck of the various flasks.

5.11 Analytical balance, of 0,05 mg accuracy.

5.12 Non-ventilated drying oven, capable of maintaining a temperature of 80 °C.

5.13 Alpha-ray ioniser, to prevent the collection of dust during the weighing operation, placed under the balance scale incorporating the filter and projecting from beneath it.

5.14 Air drier.

6 Rinsing and cleaning chemicals

6.1 Distilled or demineralized water.

6.2 Isopropyl alcohol, acetone free.

6.3 Trichlorotrifluoroethane (F. 113) ($\text{CClF}_2 - \text{CCl}_2\text{F}$) (or equivalent), or **petroleum ether**.

6.4 Liquid detergent, without solid residue.

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

Clean the filtration apparatus (5.1) and the flasks (5.9) as follows:

- wash the glassware in hot water with an admixture of liquid detergent (6.4);
- rinse three times with distilled or demineralized water (6.1);
- rinse three times with filtered isopropyl alcohol (6.2) to remove water;
- rinse three times with filtered trichlorotrifluoroethane or petroleum ether (6.3), and
 - for the filtration apparatus, turn the funnel upside down for 15 s to allow drainage and evaporation of the solvent;
 - for the flasks, leave a little solvent at the flask bottom and cover the flask by inserting a plastic film (5.10) (rinsed with the filtered solvent) between the neck and the cap.

NOTE 1 The solvent evaporation slightly pressurizes the flask and therefore avoids contamination when the flask is opened.

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 100 ml of fluid from an operating hydraulic fluid power system, by the method described in ISO 4021.

NOTE 2 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 Procedure

9.1 Filter membrane calibration

9.1.1 Take two filter membranes (5.3) out of their packings using the tweezers.

Mark them using a ball-point pen with the letters E (test) and T (gauge).

Place both membranes on the filter holder (5.1), with membrane T under membrane E. Install the funnel and fix the whole assembly by means of the clamping device.

Rinse the funnel with sufficient filtered solvent [(6.2) and (6.3)] to ensure that the funnel and the membranes are fully wetted.

Evacuate until the filter membranes are dry.

Remove the clamp and the funnel and cease evacuation.

9.1.2 Place the membranes side by side in a clean Petri dish (5.8).

Place the Petri dish with its lid half-open in the drying oven (5.12) at 80 °C, and leave it there for 30 min.

Then place the Petri dish in the drier (5.14) for 30 min.

9.1.3 Take membrane E and place it on the balance scale (5.11) after a passage over the ionizer (5.13).

Record mass m_E of membrane E to within 0,05 mg.

Take membrane T and place it on the balance scale after a passage over the ionizer.

Record mass m_T of membrane T to within 0,05 mg.

9.2 Blank test

For each measurement, parallel with the procedure described in 9.3 or 9.4, carry out a blank test using the same method (9.3 or 9.4) and deleting the fluid to be analysed.

9.3 Double-membrane method

9.3.1 Place both filter membranes in the filter holder using the tweezers, membrane T being placed in the lower position. Install the funnel and fix the whole assembly by means of the clamping device.

Stir the flask (5.9) containing the sample and remove its cap.

Remove the funnel cap and pour the flask contents into the funnel.

Pour approximately 50 ml of filtered solvent into the flask, stir and pour into the funnel.

Replace the funnel cap.

Evacuate until approximately 2 ml of fluid is left in the funnel.

Remove the cap, rinse the funnel side walls with filtered solvent, and replace the cap.

Evacuate until the membranes are dry.

Remove the cap, the clamping device and the funnel.

While under vacuum, rinse the upper membrane surface with concentric jets of solvent from the filtered-solvent dispenser (5.6).

It is necessary to use 500 ml or more of filtered solvent.

NOTE 3 The purpose of this operation is to collect sediments in the membrane middle and to perfect the gauge filter rinsing.

Cease evacuation.

9.3.2 Carry out drying operations in accordance with 9.1.2.

9.3.3 Take membrane E and place it on the balance scale after a passage over the ionizer.

Record the new mass M_E of membrane E to within 0,05 mg.

Take membrane T and place it on the balance scale after a passage over the ionizer.

Record the new mass M_T of membrane T to within 0,05 mg.

9.4 Single-membrane method

9.4.1 If a level of confidence in the calibration and validation of the membranes is evident as a result of calibration as in 9.1, the following alternative procedure may be adopted.

9.4.2 Place a single membrane in the filter holder using the tweezers. Install the funnel and fix the whole assembly by means of the clamping device.

Stir the flask containing the sample and remove its cap.

Remove the funnel cap and pour flask contents into the funnel.

Pour approximately 50 ml of filtered solvent into the flask, stir and pour into the funnel.

Replace the funnel cap.

Evacuate until approximately 2 ml of fluid is left in the funnel.

Remove the cap, rinse the funnel side walls with filtered solvent, and replace the cap.

Evacuate until the membrane is dry.

Remove the cap, the clamping device and the funnel.

While under vacuum, rise the upper membrane surface with concentric jets of solvent from the filtered-solvent dispenser.

NOTE 4 The purpose of this operation is to collect sediments in the membrane middle and to perfect the gauge filter rinsing.

Cease evacuation.

9.4.3 Place the membrane in a clean Petri dish.

Place the Petri dish with its cover half-open in the drying oven at 80 °C, and leave it there for 30 min.

Then place the Petri dish in the drier for 30 min.

9.4.4 Take the membrane E and place it on the balance scale after a passage over the ionizer.

Record the new mass M_E of the membrane E to within 0,05 mg.

10 Test results

10.1 The solid impurity content of the sample, expressed in milligrams per 100 ml of fluid, is given by the formulae

$(M_E - m_E) - (M_T - m_T)$ for method 9.3, and

$(M_E - m_E)$ for method 9.4.

NOTE 5 A difference $M_T - m_T$ greater than 0,5 mg indicates that the gauge filter membrane has not been rinsed adequately. In this case, the test should be repeated and the volume of filtered solvent to be used for rinsing should be increased.

Calculate in the same way the impurity content of the blank test (see 9.2) and, if the result is greater than 0,5 mg, subtract it from the test result.

10.2 Declare in the statement of test results the membrane pore size and the procedure utilised.

11 Test reproducibility

Compare two measurements on the same sample made by the same operator. If they differ by more than 3,6 % (m/m) of contaminant per 100 ml of fluid, repeat the operation.

12 Identification statement (Reference to this International Standard)

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

"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.*"

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