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Road vehicles — Cleanliness of components of fluid circuits —

Part 6:
Particle mass determination by gravimetric analysis

Véhicules routiers — Propreté des composants des circuits de fluide —

Partie 6: Détermination de la masse de particules par analyse gravimétrique

Reference number
ISO 16232-6:2007(E)

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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 2.

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

ISO 16232-6 was prepared by Technical Committee ISO/TC 22, *Road vehicles*, Subcommittee SC 5, *Engine tests*.

ISO 16232 consists of the following parts, under the general title *Road vehicles — Cleanliness of components of fluid circuits*:

— *Part 1: Vocabulary*

— *Part 2: Method of extraction of contaminants by agitation*

— *Part 3: Method of extraction of contaminants by pressure rinsing*

— *Part 4: Method of extraction of contaminants by ultrasonic techniques*

— *Part 5: Method of extraction of contaminants on functional test bench*

— *Part 6: Particle mass determination by gravimetric analysis*

— *Part 7: Particle sizing and counting by microscopic analysis*

— *Part 8: Particle nature determination by microscopic analysis*

— *Part 9: Particle sizing and counting by automatic light extinction particle counter*

— *Part 10: Expression of results*

Introduction

The presence of particulate contamination in a fluid system is acknowledged to be a major factor governing the life and reliability of that system. The presence of particles residual from the manufacturing and assembly processes will cause a substantial increase in the wear rates of the system during the initial run-up and early life, and may even cause catastrophic failures.

In order to achieve reliable performance of components and systems, control over the amount of particles introduced during the build phase is necessary, and measurement of particulate contaminants is the basis of control.

The ISO 16232 series has been drafted to fulfil the requirements of the automotive industry, since the function and performance of modern automotive fluid components and systems are sensitive to the presence of a single or a few critically sized particles. Consequently, ISO 16232 requires the analysis of the total volume of extraction liquid and of all contaminants collected using an approved extraction method.

The ISO 16232 series has been based on existing ISO International Standards such as those developed by ISO/TC 131/SC 6. These International Standards have been extended, modified and new ones have been developed to produce a comprehensive suite of International Standards to measure and report the cleanliness levels of parts and components fitted to automotive fluid circuits.

INTERNATIONAL STANDARD ISO 16232-6:2007(E)

Road vehicles — Cleanliness of components of fluid circuits —

Part 6:
Particle mass determination by gravimetric analysis

1 Scope

The purpose of this part of ISO 16232 is to determine the mass of particulate contamination by gravimetric analysis. Contaminants are separated from the extraction liquid by filtration. It applies to automotive components in contact with a fluid.

Unless otherwise specified, this part of ISO 16232 deals with particulate cleanliness only. It does not, therefore, cover appearance defects or contamination by liquid or gaseous materials. It covers the amount of residual particulate contaminants resulting from manufacturing processes and from the environment.

2 Normative references

The following referenced documents are indispensable for the application 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 16232-1, *Road vehicles — Cleanliness of components of fluid circuits — Part 1: Vocabulary*

ISO 16232-2, *Road vehicles — Cleanliness of components of fluid circuits — Part 2: Method of extraction of contaminants by agitation*

ISO 16232-3, *Road vehicles — Cleanliness of components of fluid circuits — Part 3: Method of extraction of contaminants by pressure rinsing*

ISO 16232-4, *Road vehicles — Cleanliness of components of fluid circuits — Part 4: Method of extraction of contaminants by ultrasonic techniques*

ISO 16232-5, *Road vehicles — Cleanliness of components of fluid circuits — Part 5: Method of extraction of contaminants on functional test bench*

ISO 16232-10, *Road vehicles — Cleanliness of components of fluid circuits — Part 10: Expression of results*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16232-1 apply.

4 Principles

The total amount of extraction liquid used in extracting particulate contaminants from components in ISO 16232-2, ISO 16232-3, ISO 16232-4 or ISO 16232-5 is filtered under either vacuum or pressure through one or more membrane filters. The number of filters is dependent on the mass of extracted contaminants. The mass difference in the membrane filter before and after the test is used to determine the mass of contaminants in the component.

5 Equipment and rinsing liquids

5.1 Membrane filters, 47 mm diameter or more, with a mean flow pore size (MFPS) of 5 µm or different provided agreed on between parties, and made of a material compatible with the liquid to be analysed. The pore size shall be clearly stated in the test report.

5.2 Membrane filter holder assembly suitable for the diameter of membrane filter chosen in 5.1. It can be made out of borosilicate, stainless steel or a suitable plastic.

WARNING — An earthing strap may have to be connected to the clamping device to dissipate any static charge generated during the filtering process.

NOTE In the ISO 16232 series, the words earthing and grounding are synonymous.

5.3 Vacuum flask of a volume suitable to hold the extraction liquid volume (e.g. 1 000 cm^3) and with a fitting to a vacuum source.

5.4 Non-magnetic flat tweezers, able to handle membrane filter without damaging them.

5.5 Petri dishes with covers to contain the membrane filter being used (see 5.1). These will normally be made of glass.

5.6 Precision balance with an accuracy of ± 0,1 mg or better (see 6).

NOTE As an example: balances at ± 0,05 mg and ± 0,1 mg are adequate, a balance at ± 0,2 mg is not adequate.

5.7 Controllable, non recirculatory oven capable of maintaining a temperature of 80 °C ± 5 °C or higher.

WARNING — Ensure that the membrane filters and any other material used in the oven contains negligible amounts of flammable liquids and the oven is operated below the flash point of any vapours.

5.8 Device for obtaining a vacuum of approximately 85 kPa (gauge).

5.9 Desiccator containing desiccant substance.

NOTE The desiccator should be placed close to the balance.

5.10 Rinsing liquid dispenser, with a suitable filter directly fitted at the outlet or filled with at least 0,8 µm prefiltered liquid and/or wash bottle (e.g. 1 000 cm^3), clean, rinsed and filled with filtered (e.g. 0,8 micron) rinsing liquid.

5.11 All liquids used in the extraction and analysis process shall be compatible with the membrane filters and all the equipment materials.

The liquids shall be sufficiently cleaned to not have a significant effect on the results

NOTE Details on equipment used should be agreed on between parties and reported in the inspection document. An example of a data sheet is given in Annex B.

6 Environment conditions and precautions

6.1 Work environment

The cleanliness of the environment where the analysis is performed shall be consistent with the presumed cleanliness of the component to be tested. This is validated when performing the blank test (e.g. according to ISO 16232-2, ISO 16232-3, ISO 16232-4 or ISO 16232-5).

NOTE Attention is drawn to the influence of humidity variations on the accuracy of the results.

6.2 Health and safety

6.2.1 General Local Health and Safety procedures shall be followed at all times, any equipment shall be operated in accordance with the manufacturer's instruction and personal protection equipment used where appropriate.

6.2.2 Chemicals used in the procedures can be harmful, toxic or flammable. Good laboratory practices shall be observed in the preparation and use of these chemicals. Care shall be taken to ensure compatibility of the chemicals with the materials used (refer to each Material Safety Data Sheet [MSDS]). Follow the precautions for safe handling and usage as described in the MSDS available from the supplier.

6.2.3 Volatile liquids: Care shall be taken with flammable liquids to ensure that they are used in accordance with the MSDS, at temperatures below the stated flash point and away from potential sources of ignition. Appropriate precautions should be taken to avoid inhalation of fumes from these solvents. Always use suitable protective equipment.

6.2.4 Electrical: Appropriate care should be applied in the use of electrical power.

6.2.5 Static: the build-up of electro-static charges (created by friction as fluids flow) shall be dissipated and not be allowed to build- up where it can discharge and create a spark. An earthing strap shall be provided where there is a risk, especially for the vacuum where often volatile liquids are involved.

6.2.6 Disposal all liquids and substances shall be disposed of in accordance with local environmental procedures. In the event of spillage it shall be cleaned-up in the manner detailed in the MSDS.

7 Lower detection limit for gravimetric analysis

Where objects are weighed using balance with an accuracy of at least ± 0,1 mg in rooms with uncontrolled conditions as far as temperature and relative humidity are concerned, the lower detection limit is 1 mg. With a blank requirement of less than 10 % of the final mass, the residual mass (m_F) shall be at least 10 mg in order for it to be accurately weighed.

In practice, the possibility to analyse gravimetric levels below 10 mg in an uncontrolled environment should not be excluded. In this case, blank values below 1 mg are acceptable to analyse a contaminant mass between 3 mg and 10 mg. Report the blank value as being "< 1 mg".

When using a five or six digit balance with an accuracy of at least 0,01 mg in rooms with controlled temperature and relative humidity, it is possible to detect blank values $<$ 1 mg. For this reason, analysis of a contaminant mass of $<$ 10 mg respecting the 10 % criterion is possible.

For the case of very low gravimetric level, another alternative method of inspecting components could be to increase the residual mass (m_F) by performing extraction on several components simultaneously or by filtering the analysis liquid collected from several components through one membrane filter.

8 Preparing the apparatus

8.1 Conditioning of equipment

Prepare clean filtration equipment, membrane filter holders, collection and rinsing vessels, tweezers and analysis equipment. The required cleanliness level of the equipment shall be such that the equipment will not contribute significantly to the overall result and shall be consistent with the presumed cleanliness of the component to be tested.

8.2 Preparing the membrane filters

8.2.1 Remove one membrane filter (see 5.1) from its packaging using the tweezers (see 5.4) and carefully rinse its two sides with rinsing liquid.

NOTE Rinsing the membrane filter is optional when a high weight of contaminant is presumed to be measured.

8.2.2 Place the membrane filter on its holder (see 5.2).

8.2.3 Apply vacuum until the membrane filter is dry.

8.2.4 Release the vacuum, remove the membrane filter and place it in a Petri dish (see 5.5).

8.2.5 Place the Petri dish with its cover ajar in the oven (see 5.7) for the minimum period of time at the minimum temperature as determined in the validation procedure (e.g. 30 min at 80°C). During this operation ensure that no contamination reaches the membrane filter.

NOTE The drying time is dependant on the volatility of the rinsing liquid, and may have to be determined individually. The drying time can be reduced by using a vacuum desiccator.

8.2.6 Place the Petri dish with its cover kept ajar in the desiccator (see 5.9) for the minimum period of time as determined in the validation procedure.

NOTE In a controlled environment (temperature and humidity) the use of a desiccator is optional.

8.2.7 Remove the membrane filter from the Petri dish and weigh it immediately on the balance to within 0,10 mg. Note its mass (m_1) and return the membrane filter to the Petri dish.

9 Procedure

9.1 Evaluation of the procedure

Depending on a specific combination of extraction liquid and type of membrane filter used, the following applies:

a) Appropriate parameters and settings of temperature and duration of the drying procedure shall be established individually. Record this data on the record sheet and/or in the report.

b) It should be known if the liquids used possibly affect the weight of the membrane filter due to dissolving the membrane filter material.

c) It should be known if the components under test contain any non-dissolvable chemical substances which might affect the residual mass on the membrane filter, e.g. grease, preservative fluids.

NOTE The presence of such chemical substances will cause errors and variability in data, and test components containing it should be excluded from the analysis. However, if it is essential to analyse the item, then the extraction liquid and rinsing liquid used to wash the membrane filter are chosen so that it completely dissolves the chemical substance.

9.2 Liquid analysis procedure

9.2.1 Place the membrane filter (see 5.2) on its holder and securely clamp the membrane filter. If applicable, connect earthing strap to the clamp.

9.2.2 Vacuum filter the total amount of extraction liquid (do not apply vacuum greater than 80 kPa).

9.2.3 Rinse the collection equipment thoroughly to remove all residual contaminants.

9.2.4 Immediately after filtration, carefully rinse the funnel thoroughly with rinsing liquid (see 5.11) using the wash bottle or pressure dispenser (see 5.10).

9.2.5 With the vacuum still applied (if necessary), remove the clamping device and funnel, rinse the edges of the membrane filter thoroughly with the rinsing liquid using a circular movement from the outside towards the centre.

This operation is intended to remove all traces of liquid from the membrane filter and to move contaminants toward the center of the membrane. It shall be carried out carefully, ensuring that all contaminants remain on the surface of the membrane filter.

9.2.6 Leave the membrane filter to dry and gradually release the vacuum.

9.2.7 Remove the membrane filter and place it in a Petri dish (see 5.5).

9.2.8 Place the Petri dish and its cover in the oven (see 5.7) for the minimum period of time and at the minimum temperature as determined during the evaluation of the procedure (see 9.1) (e.g. 30 min at 80 °C). Record these data on the record sheets and/or in the report.

NOTE The drying time is dependant on the volatility of the rinsing liquid, and may have to be determined individually. The drying time can be reduced by using a vacuum desiccator.

9.2.9 If necessary, place Petri dish, its cover and membrane filter in the desiccator (see 5.9) for the minimum period of time as determined during the evaluation of the procedure (e.g. 30 min) before weighing.

9.2.10 Remove the membrane filter from the Petri dish and weigh it immediately on the balance (to within e.g. 0,10 mg). Note its mass (m_2) and return the membrane filter to the Petri dish.

10 Reporting the results

10.1 Calculating the mass of contaminants

The contaminant mass found on the component (m_{Cc}) is given by the sum of the masses of contaminants ($m_{\mathrm{C}i}$) found on each membrane filter, which is calculated using the relation $m_{\mathrm{C}i} = m_2 - m_1$.

$$m_{\mathrm{Cc}} = \frac{1}{p}\sum_{i=1}^{n} m_{\mathrm{C}i}$$

where

n is the number of membranes necessary to filter the whole extraction liquid volume;

m_1 is the mass of clean membrane filter as determined in 8.2.7;

m_2 is the mass of membrane filter with contaminants as determined in 9.2.10;

p is the number of components analysed.

10.2 Reporting of the level of contamination of the component

Calculate the gravimetric contamination level (m_{CON}) of the component by dividing the mass of contaminants m_{Cc} by the relevant unit (i.e. wetted volume, wetted surface, number of components).

Report the results of the component cleanliness examination as per ISO 16232-10.

If the actual measured value is less than 3 µg, then report “mass weighed < 3 µg”.

NOTE Using a four digit balance, under uncontrolled, environmental conditions (uncontrolled humidity and temperature), the minimum measurable mass is 0,3 mg.

11 Test report

In addition to the results obtained, the test report shall indicate at least:

- the reference of this method;
- the address of Company/Laboratory performing the analysis;
- the report number and date;
- the date of analysis;
- the test component name, identification number and quantity;
- the type of test liquid used;
- the balance reference number and details;
- the membrane filter diameter and pore size;
- the blank results;
- operating details not included in the method and any incident that may have affected the results.

An example of test report is given in Annex A.

Annex A
(informative)

Example of standard gravimetric analysis report as per ISO 16232-6

A.1 Customer identification

Company: ______________________ Order: ______________________
Contact: ______________________ Date: ______________________
Address: ____

A.2 Report and analysis identification

Laboratory identification __________ Report Number: ______________ Project No. ______
Date of analysis: ____________________ Operator: ______________________

A.3 Test component identification

Type: ______________________ Reference: ______________
Initial conditioning: ______________________ Serial Number: ______________
Number of parts analysed: ______________________
Wetted surface area: ______________ cm^2 Wetted volume: ____________ cm^3
Controlled surface area: ______________ cm^2 Controlled volume: ____________cm^3
Material: ______________________

A.4 Operating conditions

A.4.1 Extraction procedure

Method: ______________________ Reference Number: ______________
Liquid: Type: ______________________ Volume: ______________
Environment: ☐ Industrial ☐ Laboratory ☐ Controlled (ISO 14644-1 Class: ________)

A.4.2 Analysis equipment

Rinsing liquid: Type: ______________________
Membrane filter: Material: __________ Diameter: mm Mean pore size: ________ µm
Balance: Reference: ______ Date of last calibration: ________ Range: ______ Accuracy: ______ mg
Environment: ☐ Industrial ☐ Laboratory ☐ Controlled (ISO 14644-1 Class: ________)

A.5 Test results

Volume of sample: ____________ mL filtered on n = _________ membrane filter(s)

	Test 1	Test 2	...	Test n
Blank (mg)				
Membrane + contaminant m_2 (mg):				
Membrane m_1 (mg):				
Mass of contaminant $m_{Ci} = m_2 - m_1$ (mg):				
Total mass of contaminants collected: $m_{Cc} = \sum_{i=1}^{n} m_{Ci}$ = …..mg				
Mass per component / 1000 cm^2 / 100 cm^3 [a]: m_{CON} = ———— mg/———— [b]				

[a] Score out unused parameter.

[b] Report appropriate parameter.

A.6 Observations/comments

Date:

Name:

Signature

Annex B
(informative)

Filtration

B.1 Filtration connected to the extraction equipment

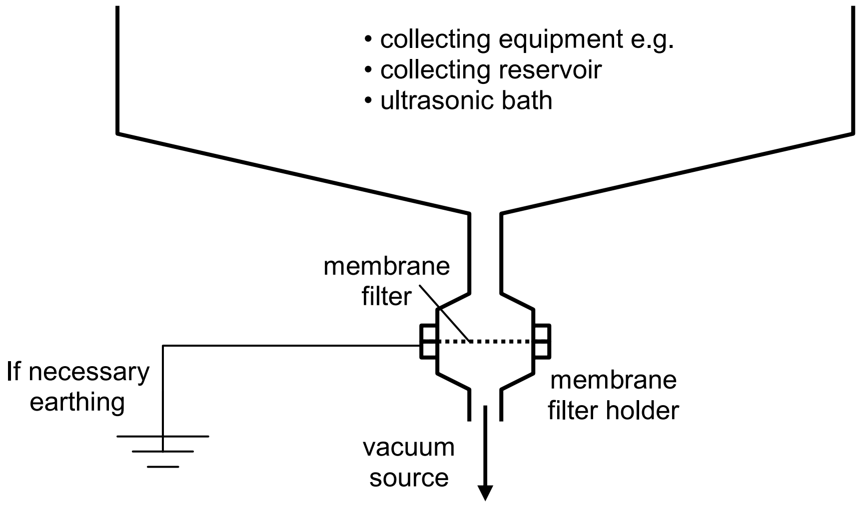

Figure B.1 — Filtration connected to the extraction equipment

B.2 Separate filtration

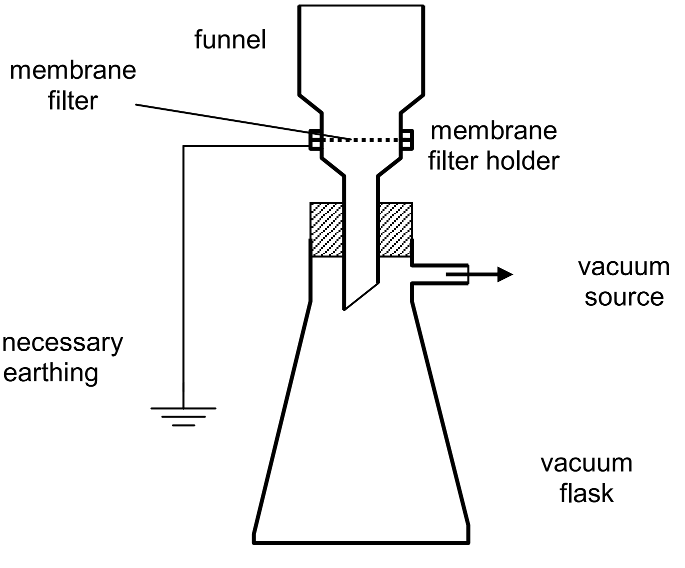

Figure B.2 — Separate filtration

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