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Road vehicles — Cleanliness of components of fluid circuits —

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

Véhicules routiers — Propreté des composants des circuits de fluide —

Partie 9: Granulométrie et comptage des particules au moyen d'un compteur de particules automatique à extinction de la lumière

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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 16232-9 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 of 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 contamination 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.

This part of ISO 16232 defines a method of automatic counting to determine the particle size distribution of contaminants which have been removed from the component under test and collected using an approved extraction method.

Road vehicles — Cleanliness of components of fluid circuits —

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

1 Scope

This part of ISO 16232 defines methods for determining the size distribution of particulate contaminants extracted from automotive components using techniques described in ISO 16232-3, ISO 16232-4 and ISO 16232-5, using automatic light extinction particle counter instruments (APC).

It only applies to the analysis of the whole volume of extraction liquid.

The size range of particles that can be measured by this technique is limited to ⩽ 70 µm(c), because of the method of calibration used (ISO 11171). However, the calibration can be extended to other sizes provided that it is agreed and included in the Inspection Document.

This technique is only applicable to measuring particles contained in clear, single phase liquids.

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 11171, *Hydraulic fluid power — Calibration of automatic particle counters for liquids*

ISO 11943, *Hydraulic fluid power — On-line automatic particle-counting systems for liquids — Methods of calibration and validation*

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 16232-7, *Road vehicles — Cleanliness of components of fluid circuits — Part 7: Particle sizing and counting by microscopic analysis*

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 Principle

Extraction samples are passed through the sensing volume of an APC where particles are sized and counted based on the calibration of the instrument with particles of defined properties. The primary result obtained is the quantitative particle size distribution of the contaminants contained in the analysed sample, either differential and/or cumulative.

The analysis procedure consists of the following basic steps:

a) cleaning the analysis equipment;

b) conditioning the extraction liquid;

c) analysis of the extraction liquid and analysis of a subsequent rinsing liquid;

d) expression of results.

NOTE 1 Further information on APCs is given in Annex A.

NOTE 2 An example of the procedure is given in Annex B.

Immediately after the extraction sample has been analysed, a subsequent sample of clean rinsing liquid shall be analysed likewise in order to characterise all detectable particles remaining in the analysis set up. The particle counts from the subsequent sample shall be added to the counts obtained from the extraction sample.

NOTE 3 For correct operation of the APC, there is only one particle in the sensor at any one time, otherwise errors from particle coincidence will be experienced.

The particles are sized in terms of the amount of light reduced by the passage of the particle through the light beam, and the relationship between extinction signal and size is obtained by calibration. It is referenced to the equivalent spherical diameter of calibration material. The measurement principle is seen in Figure 1.

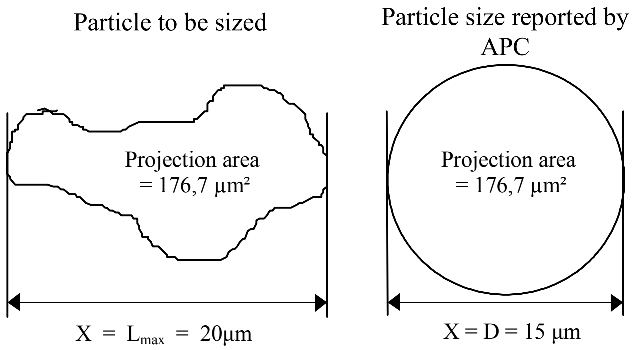

Figure 1 — Particle sizing by APC based on diameter of equivalent projection area

During analysis, all care shall be taken not to degrade or additionally contaminate the extraction sample and not to loose any particle e.g. due to sedimentation or adhesion. As the function of parts and components can be impaired due to single or a few critical particles, a complete analysis of the total volume of the extraction sample is essential.

5 Equipment

5.1 General

All equipment for the analysis shall be sufficiently clean so as not to affect the results of the analysis. This is validated by the blank analysis (see 7.3).

5.2 Liquids

Liquids are used to both clean and rinse the equipment before and after analysis, and for diluting the extraction liquid prior to analysis. All liquids used shall be totally miscible with one another if used sequentially and shall be compatible with the equipment used.

The liquids shall be cleaned by filtering prior to use to give an appropriate cleanliness level.

Only clear single phase liquids without interface and with low viscosity (aqueous liquids or solvents) may be used. The refractive index of the liquids shall be similar to the one of the liquid used for calibration of the sensor.

NOTE Use of liquids with a refractive index different from the refractive index of the liquid used for calibration may cause counting and sizing errors.

5.2.1 Diluent and rinsing liquid

A suitable liquid for diluting the sample and for rinsing the analysis set-up shall be filtered through a membrane filter or a suitable cartridge filter (e.g. 0,45 µm). The refractive index of the liquid shall not differ from that of the extraction liquid by more than 10 %.

The cleanliness level of the liquid shall be checked and validated before use. This is validated when performing the blank test of the set-up (e.g. according to ISO 16232-2).

5.2.2 Clean-up filter

Clean-up filters for liquids are chosen to achieve the required cleanliness level and have a retention capacity such that it does not require replacement too often in order not to introduce particles.

5.2.3 Liquid dispensers

Liquid dispensers, fitted with a suitable membrane filter directly at the outlet or filled with pre-filtered liquid.

5.2.4 Filtration apparatus

Filtration apparatus, suitable for filtering the various liquids used in the procedure.

5.3 Sample containers

The containers are normally cylindrical glass or polypropylene bottles, fitted with either a suitable nonshedding threaded cap forming a seal with the bottle without the use of an insert, or a cap with a suitable internal seal. The bottle should be flat-bottomed and wide-necked to facilitate cleaning.

Sample containers (bottles) shall be cleaned so as to fulfil the blank requirement.

These containers can be used for transportation and storage of the extraction liquid (if required) and in the dilution of extraction samples.

5.4 Additional equipment

5.4.1 Analysis reservoir

Conical shaped container, equipped with a non-shedding stirrer, to hold the liquid sample and serve as the supply reservoir for the APC and sampling apparatus. It also serves to ensure that contaminants are put in homogeneous suspension and air bubbles are removed prior to analysis.

NOTE The outlet for the APC should preferably be located at the lowest point of the bottom of the reservoir.

The stirrer chosen shall not modify the size distribution of the extracted particles.

5.4.2 Sampling device

Pump or similar device and related pipework used for directing the liquid through the sensor of the APC. Examples of configurations are given in Annex C.

NOTE For any combination of APC and sampling apparatus used, it is safeguarded that even a single large sized particle contained in the analysis reservoir can be fed to the sensor of the APC.

5.4.3 Stirrer

Non-magnetic device with adjustable speed, integrated into the analysis reservoir.

A magnetic stirrer in the analysis set-up shall not be used for samples containing ferrous or other magnetic particles. If such a stirrer is fitted as standard equipment, remove or negate the drive magnet.

5.4.4 Flowmeter

Calibrated device mounted downstream from the sensor to measure the extraction sample flow rate. Its accuracy shall match the flow regulation required by the sensor, and shall be stated by the APC manufacturer. A flowmeter shall be required if a controlled syringe is not used (see Annex C).

5.4.5 Device for degassing (optional)

Device for degassing the liquid samples, e.g. vacuum reservoir or ultrasonic bath.

5.5 Analysis equipment

5.5.1 Automatic light extinction particle counter (APC)

A device based on the light extinction principle consisting of a sensor to detect the particles within the specified size range and an instrument to size and count the electrical signals generated by the sensor as the particles pass through.

The APC shall be capable of providing non-coded data representing the raw counts of particles of the sizes specified in the inspection document that are detected during analysis.

As the function of parts and components can be impaired due to single or a few critical particles, the sensing volume of the APC shall be able to analyse 100 % of the stream of liquid flowing through the sensor.

The instrument shall be used in accordance with the instrument manufacturer's recommendations. All measurements shall be made at particle concentrations which are below 80 % of the instrument manufacturer's stated coincidence limit and at a size which is at least 1,5 times above the "noise" level of the instrument.

NOTE 1 Coincidence causes an overcount of larger particles and an undercount of smaller particles. The coincidence limit of the counter is the maximum acceptable concentration of all the particles detected by the instrument. This concentration is normally given by the instrument manufacturer with a note indicating the probability of coincidence. Coincidence is reduced by dilution.

NOTE 2 The “noise” level of the instrument is the minimum voltage setting of the detection circuit below which spurious electrical signals become significant and are counted as particles.

The sensing volume should be inspected for the presence of particles on a regular basis, either in the sensing volume itself or in the entry to it.

5.5.2 Data acquisition unit

Device for recording of the data provided by the APC.

5.5.3 Measuring range

The measuring range depends on the calibration and type of APC. The dynamic size range of the APC used should meet size ranges of the cleanliness specification or the presumed cleanliness size ranges of the component under test.

The setting of size intervals of the sensor shall be selected so as to cover the particle size classes specified in the inspection document following the size classes according to ISO 16232-10.

NOTE For example see E.2.

5.6 Environment conditions and precautions

5.6.1 Work environment

The cleanliness of the environment where the analysis is performed shall be consistent with the presumed cleanliness of the component under test. This is validated when performing the blank test.

5.6.2 Electrical interference

As the APC is typically a high-sensitivity device, it may be affected by radio frequency interference (RFI) or electromagnetic interference (EMI). Precautions shall be taken to ensure that the test area does not exceed the RFI and EMI capabilities of the instrument.

In addition, the voltage supply to the instrument shall be stable and free of excessive noise which affects the operation of APC (see 5.5.1 and 5.6.4). A constant voltage transformer is considered appropriate.

5.6.3 Chemicals

Chemicals used in the procedures can be harmful, toxic or flammable. Good laboratory practices should be observed in their preparation and use. Care shall be taken to ensure chemical compatibility with the materials used.

Temperature of test samples shall not exceed temperature limitations of the test equipment.

5.6.4 Influencing parameters

APC operation can be affected by bubbles and non-homogeneous liquids as well as by the environmental conditions indicated in 5.6.1 to 5.6.3.