INTERNATIONAL STANDARD

ISO 16232-8

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Road vehicles — Cleanliness of components of fluid circuits —

Part 8: Particle nature determination by microscopic analysis

Véhicules routiers — Propreté des composants des circuits de fluide — Partie 8: Détermination de la nature des particules par analyse microscopique

Reference number
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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 16232-8 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 liquid 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.

This part of ISO 16232 defines a method of microscopic examination to determine the nature of contaminants which have been removed from the component under analysis and collected using an approved extraction method. It can be used at the same time to determine the particle size distribution as described in ISO 16232-7.

Road vehicles — Cleanliness of components of fluid circuits —

Part 8:
Particle nature determination by microscopic analysis

1 Scope

This part of ISO 16232 describes a method for determining the nature of contaminant particles by identifying their elemental chemical composition using energy dispersive X-ray spectroscopy (EDX) in combination with a scanning electron microscope (SEM). The contaminant particles are extracted from automotive parts or components and deposited on the surface of a membrane filter. In addition to the number and size of particles as described in ISO 16232-7, this measurement gives the elemental composition of the particles analysed.

This information can be used to classify the particles into likely material groups [1].

This method cannot determine the nature of organic material [2].

The analyses can either be carried out manually or fully automatically, provided the appropriate equipment is available.

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 15632:2002, *Microbeam analysis — Instrumental specification for energy dispersive X-ray spectrometers with semiconductor detectors*

ISO 16232-1, *Road vehicles — Cleanliness of components of fluid circuits — Vocabulary*

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

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

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

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

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1) The additional material information can be used to give a more detailed characterization of particles or to find their source in production process. The range of elements detected by the SEM/EDX system depends upon the design and configuration of the separate items.

2) Infrared or other techniques can be used to detect organic particles.

ISO 16232-7:2007, *Road vehicles — Cleanliness of components of fluid circuits — Particle sizing and counting by microscopic analysis*

ISO 16232-10:2007, *Road vehicles — Cleanliness of components of fluid circuits — 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 entire volume of extraction liquid used to extract particles from the test component (as described in ISO 16232-2, ISO 16232-3, ISO 16232-4 and ISO 16232-5), is filtered and the separated particles are counted and sized using a SEM according to 16232-7. In the process, the sample to be imaged is scanned point-for-point in a vacuum using a finely-focused high-accelerated electron beam. This treatment with high-energy electrons leads the sample to give off X-rays that are characteristic of the chemical elements in the particle being examined. By recording this spectrum using an EDX detector, it is possible to determine the elements in the residual contamination particles. The analysis of the intensity of the spectral lines also enables a quantification of the elements concerned. The result is the chemical composition of the particles investigated.

Particles are located on the surface of the membrane filter using the Back Scattering Electron (BSE) detector of the SEM and an element analysis is then carried out only at these points.

5 Equipment

5.1 General

All the equipment that is used for preparing the membrane filter or the components and software features of a SEM suitable for particle analysis is described in ISO 16232-7.

5.2 Analysis equipment

5.2.1 Principle

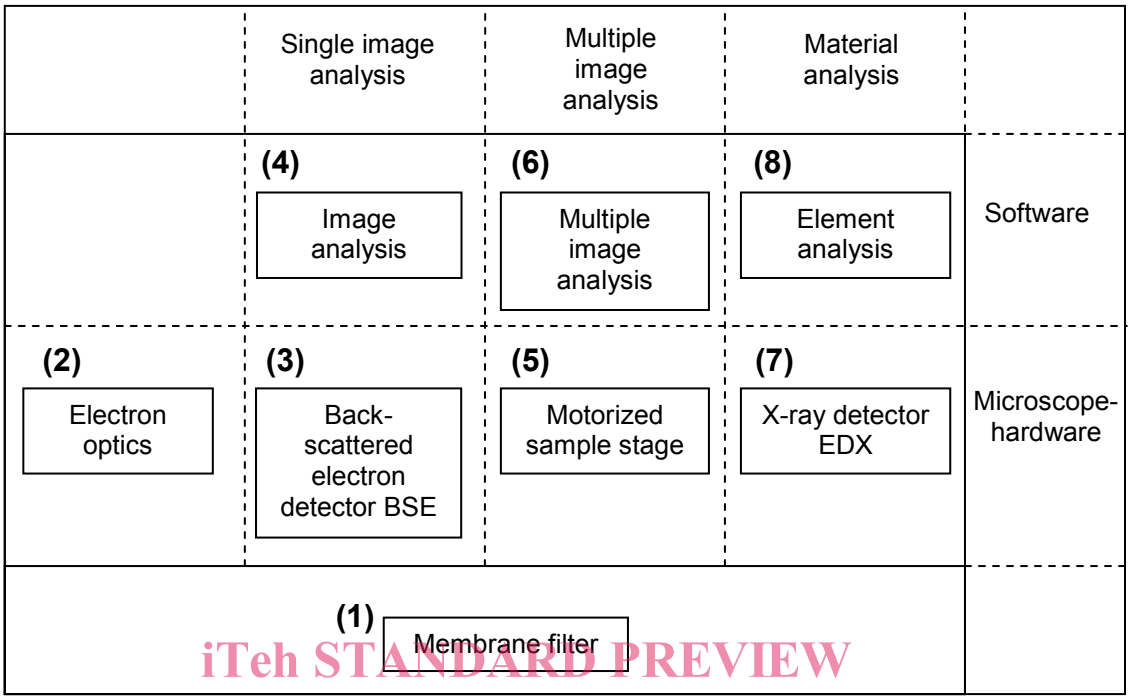

Figure 1 — Diagrammatic representation of the membrane filter analysis using a SEM equipped with an EDX system used for element analysis

As shown in Figure 1, the principle set-up of this analysis and the device technology used are almost identical to those implemented for particle sizing and counting using an SEM (see 5.2 of ISO 16232-7:2007). For the element analysis, an additional detector (X-ray or EDX detector) (7) and the corresponding element analysis software (8) are required. For electron-optical systems, further requirements exist.

5.2.2 Electron optics

The stability of the electron beam current is crucial to the quality of the analysis. As a reference, a deviation of approximately 1% per hour in the strength of the beam is acceptable. This is measured either by a Faraday cup which is introduced into the electron beam or from the counting rate of the EDX detector on an element standard.

The cathode which generates the electrons shall be warmed until its emissions are stable.

NOTE The cathode types of tungsten, LaB_6 and hot field emitters are all suitable for these measurements. However, devices with cold field emitters often show excessive levels of beam strength instability.

When an X-ray detector is integrated into a scanning electron microscope, it is mounted onto the vacuum chamber so that the “direction of view” of the detector cuts across the electron beam at the working distance of the microscope. In order to be able to carry out reproducible measurements, all analyses must be performed using this working distance. The working distance between the work piece and the deflector lens varies from unit to unit and will be specified by the manufacturer of the system.

5.2.3 X-ray detector

The higher the energy dispersion of the detector, the better the resolution. This means that elements having X-ray lines close together in the spectrum will be better separated and a more accurate result will be obtained.