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
ISO 16232-8:2007(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 16232-8 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.

INTERNATIONAL STANDARD **ISO 16232-8:2007(E)**

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

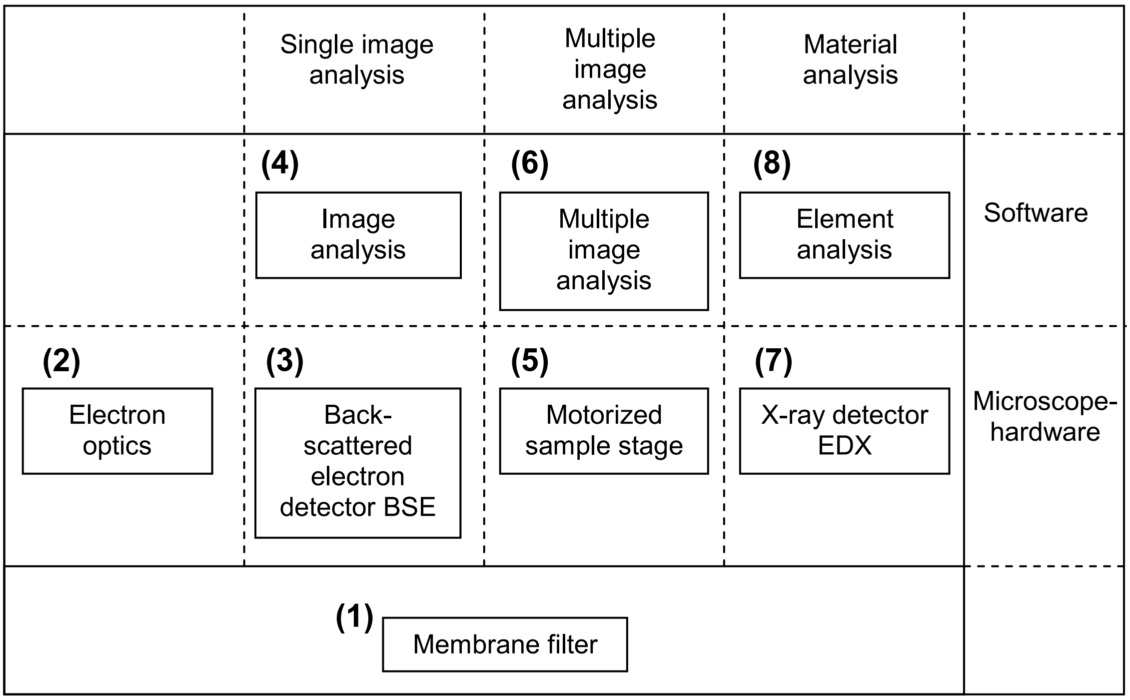


Figure 1 — Diagrammatic representation of the membrane filter analysis using a SEM equipped with an EDX system used for element analysis

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.

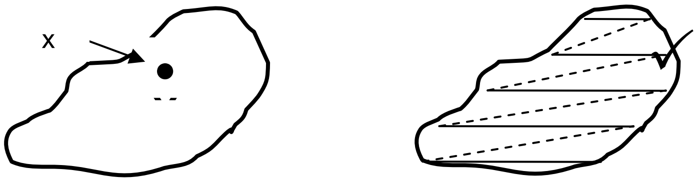
When an X-ray detector is integrated into a scanning electron microscope, it is mounted onto the vacuum chamber so that the "direction of view" of the detector cuts across the electron beam at the working distance of the microscope. In order to be able to carry out reproducible measurements, all analyses 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.

5.2.4 Element analysis

To obtain a correct result of the composition of elements, the EDX system shall be able to analyze the entire area of the detected particle. This means that the electron beam is guided to a number of measuring points over the particle during the EDX analysis. This reduces the risk which exists, when performing a measurement using only one point, that a non-homogeneity in the particle or a foreign substance adhering to it (e.g. production process materials such as oil or grease) be classified as being part of the particle itself, see Figure 2.



Fixed position, X, of electron beam: Not suitable for correct particle analysis

Suitable analysis by measuring a number of points on a particle, e.g. in a scanned pattern

Figure 2 — Example of suitable or non suitable particle analysis

To make the characterization of the particles easier, the EDX system should possess the ability to group the particles into classes based on the percentage of elements in their composition (e.g. copper in connection with zinc is classified as brass), see Classification Table in Annex A.

Particles which do not fit to any material class with regard to chemical composition shall be counted in a separate class (e.g. unclassified particles).

On completion of a fully-automated analysis, automatic systems shall be able to find individual particles, analyze them manually and, where necessary, classify them afterwards.

5.2.5 Measurement limits

The determination of a material is performed exclusively according to the composition in percent of the elements making up the particle. In order to obtain further information regarding their exact chemical bonding from the chemical shift, the energy dispersion from EDX detectors is inadequate.

Due to their lack of material contrast on a filter membrane, it is very difficult to classify organic compounds. Generally, organic compounds can be detected, but, because of their high carbon signals, cannot be classified as being made of a specific material. Exceptions to this include, for example, synthetic materials containing elements additional to carbon, oxygen and nitrogen, such as halogenated plastics (PVC, PTFE, etc.).

5.3 Environmental conditions

The cleanliness of the environment where the analysis is performed has to be adapted to the presumed cleanliness of the component to be tested. This is validated when performing the blank test.

The site for the microscope should be selected to avoid environmental factors such as vibration of the building, or external light from influencing the imaging quality and accuracy of the particle measurement. If these factors cannot be controlled, appropriate measures shall be taken (vibration absorbers, encapsulation, etc.).

5.4 Health and Safety

5.4.1 Local Health and Safety procedures shall be followed at all times, any equipment shall be operated in accordance with the manufactures instructions and personal protection equipment used where appropriate.

5.4.2 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.

5.4.3 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.

5.4.4 Electrical: appropriate care should be applied in the use of electrical power.

5.4.5 Static: the build-up of electro-static charges (created by friction as fluid 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 equipment where often volatile liquids are involved.

5.4.6 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.

6 Calibration

The EDX detector shall be calibrated and dispersion determined on completing its installation and in conjunction with the SEM. The calibration shall be carried out in accordance with the manufacturer's instructions. Pure element standards function as calibration substrates and this is described in ISO 15632:2002.

The calibration of magnification of the SEM in order to perform correct particle sizing is described in ISO 16232-7.

The calibration period depends upon the usage of the instrument, and the instrument manufacturer will advise on this. However, for single shift application, a calibration period of one year is considered appropriate. It shall be re-calibrated if the EDX system has been repaired, serviced, or adjusted. A validation procedure shall be developed and used periodically (e.g. monthly) to ensure the continued efficacy of the instrument.

7 Procedure

7.1 General

The procedure for preparation of both the SEM/EDX and the membrane filter is identical to the procedure required for particle counting using an SEM as described in ISO 16232-7 but with an additional EDX analysis described here.

7.2 Element analysis procedure

7.2.1 Analysis parameters

The parameters are set for the SEM and its components as described in ISO 16232-7:2007, 7.3, with the difference that individual parameters are selected in such a way to enable an exact and reproducible element analysis to be performed. Additional or deviating points are described below.

7.2.2 Z-position

The z-position of the sample stage (distance between the work piece and the deflector lens) shall be set so that the membrane filter is at the correct working distance.

7.2.3 Acceleration voltage

The acceleration voltage of the SEM should be adjusted to 20 kV for the element analysis. In this way, an X-ray spectrum range is available for the analyses which enables the clear separation of the spectral lines and thus of the elements.

NOTE 1 In principle, all elements can also be detected using lower voltages of acceleration, e.g. by analyzing the L or M lines of the X-ray spectrum. If done, these lines are very close to one another making a separation of the elements more difficult.

NOTE 2 With certain samples/membrane filters, due to electrical charging or thermal stress brought about by the electrons, it may be necessary to work with acceleration voltages other than 20 kV. These are mentioned in the documentation.

7.2.4 The intensity of the beam strength

The intensity of the electron beam current shall be set in such a way that the EDX system counts and analyses a number of X-ray quanta which is adequate from a statistical point of view. The higher the number of quanta detected, the more precise the analysis result will be. As a reference, a minimum of 2000 usable counting events per second (2000 cps) are recommended, independent of the type of device used. For the same reason the analysis time per detected particle should not be less than one second.

7.2.5 Sensitivity of the BSE detector

The sensitivity of the BSE detector shall be set to enable all relevant elements to be detected and to permit the dynamic range of the image analysis to be used effectively.

NOTE It is advisable that measurement is not started until both beam strength and vacuum have stabilized (allow the cathode to warm up).

8 Expression of results

The “component cleanliness reporting sheet” described in Annex C of ISO 16232-10:2007 is extended by a line below each size class specified for each material specified for characterization.

Table 1 — Example of a result with 6 material classes

Size class	B	C	D	E	F	G	H	I	J	K
µm	$5 \leqslant x$ <15	$15 \leqslant x$ <25	$25 \leqslant x$ <50	$50 \leqslant x$ <100	$100 \leqslant x$ <150	$150 \leqslant x$ <200	$200 \leqslant x$ <400	$400 \leqslant x$ <600	$600 \leqslant x$ <1 000	$1\,000 \leqslant x$
Number counted										
Low-alloy steel	- [a]	-	411	63	12	2	0	0	0	0
High alloy steel	-	-	126	24	8	2	0	0	0	0
Brass	-	-	36	11	1	0	0	0	0	0
Copper	-	-	82	9	0	0	0	0	0	0
Aluminum	-	-	12	0	0	0	0	0	0	0
Not classified	-	-	345	98	32	9	3	0	0	0

[a] A dash “-” signifies that, for this esample, data is not required.

The analysed particles are grouped into material classes based on the percentage of elements in their composition. See classification table in Annex A. This classification shall be agreed between parties.

Annex A
(informative)

Example of classifying particles according to their chemical composition

Class	**Element**	**Content %**
Steel non-alloyed	Fe	>95
	Mn	<1
	Si	<1
Steel low-alloy	Fe	>85
	Cr	<10
	Ni	<5
	Mn	<1
	Si	<2
Steel high-alloy	Fe	50-80
	Cr	10-40
	Ni	0-15
	W	1-10
	Mo	1-10
	V	1-10
Brass	Cu	20-80
	Zn	20-80
Bronze	Cu	20-80
	Sn	20-80
Cast iron		
Any others		

NOTE The classification used should be verified with samples of materials to be measured (e.g. typical alloys in a production process).

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