INTERNATIONAL STANDARD

ISO 16232-2

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Road vehicles — Cleanliness of components of fluid circuits —

Part 2: Method of extraction of contaminants by agitation

Véhicules routiers — Propreté des composants des circuits de fluide —

Partie 2: Méthode d'extraction des contaminants par agitation

Reference number
ISO 16232-2:2007(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 16232-2 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/SC6. 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 procedures for the removal and collection of contaminants from components using a moving test liquid (agitation) so that their cleanliness can be evaluated.

The cleanliness level of a component, as determined according to this method, depends to a large extent on the test parameters (e.g. type of agitation, duration of agitation, choice of test liquid, etc). All parameters should be included in the cleanliness specification and in the inspection document and should be rigorously followed by the test staff.

Road vehicles — Cleanliness of components of fluid circuits —

Part 2:
Method of extraction of contaminants by agitation

1 Scope

This part of ISO 16232 describes the principles of extraction of contaminants from a component by the agitation method. It is preferably applied to components that are hollow and are suited to being agitated by an operator or by an appropriate mechanical device.

This agitation method can be employed on its own or in association with other methods of extraction described in the ISO 16232 series.

Unless otherwise specified, this part of ISO 16232 deals with particulate contamination only. It does not, therefore, cover appearance defects or contamination by liquid or gaseous materials. It covers the amount and the nature of residual particles 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 16232-3, *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 16232-6, *Road vehicles — Cleanliness of components of fluid circuits — Part 6: Particle mass determination by gravimetric analysis*

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

ISO 16232-8, *Road vehicles — Cleanliness of components of fluid circuits — Part 8: Particle nature determination by microscopic analysis*

ISO 16232-9, *Road vehicles — Cleanliness of components of fluid circuits — Part 9: Particle sizing and counting by automatic light extinction particle counter*

ISO 16232-10:2007, *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

The contaminants are extracted by partially filling the component with a known volume of test liquid, sealing its openings, and agitating it in order to extract the particles from the controlled surfaces and suspend them in the extraction liquid for subsequent analysis.

5 Equipment

5.1 General

The equipment used shall neither alter nor modify the size distribution of the extracted particles.

5.2 Test liquid

The test liquid shall be compatible with all the materials in the component, with the liquid used in the final system and with the test equipment, including seals, membrane filter and clean-up filter. A test liquid of low viscosity ($\leqslant 5\ mm^2/s$) and having the capability of removing (or dissolving) oil and grease is recommended. It should be filtered to attain the requirements of 6.3.3.

SAFETY PRECAUTIONS — In case a tested component will be reclaimed for final use, application of incompatible test liquid may cause hazardous damage

5.3 Test component container

A closed container should be used for the transfer of the component from the place of sampling to the place of particle extraction. This container shall be appropriate to the shape of the component and made of material compatible with the test liquid. Its degree of cleanliness shall comply with the blank requirements specified in 6.3.3.

5.4 Pressure rinsing liquid dispenser

The pressure liquid dispenser is a device that provides a clean liquid at a pressure and flow rate capable, in an effective manner, of rinsing residual contaminants from sampling equipment, collection containers, test component and analysis apparatus.

NOTE This device can be same as the one used for providing the test liquid.

5.5 Vacuum suction system

If necessary, use an assembly consisting of a source of vacuum, a vacuum flask previously cleaned and a flexible tube of suitable dimensions and shape for recovery of the extraction liquid and any particles that have accumulated in the component under examination.

5.6 Collection equipment

The collection equipment shall allow effective draining of particles. A conical base is preferred.

It shall be cleaned to achieve the requirement of 6.3.3.

It is possible for contaminants remaining on the equipment to be transferred to the sample and thus be erroneously included as part of the particles removed from the component. All collection equipment shall be cleaned and covered before use in order to limit contamination from the environment.

5.7 Sampling containers

The sampling containers (glassware, etc.) required for transferring the extraction liquid from the collection equipment to the analysis equipment shall be cleaned to achieve the requirements of 6.3.3.

5.8 Environmental conditions

The cleanliness of the environment where the extraction is performed shall be consistent with the presumed cleanliness of the component to test. This requirement may result in the test being carried out in a laboratory or controlled workplace. The suitability of the environment is validated when performing the blank test.

5.9 Health and safety

5.9.1 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.

5.9.2 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.

5.9.3 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.

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

5.9.5 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 Procedure

6.1 Handling and storage

6.1.1 During handling and storage of test components, it shall be ensured that no contaminants are deposited on or removed from controlled surfaces.

6.1.2 To prevent loss of particles during transport, it may be necessary to seal openings of the test components, e.g. with suitable plugs.

6.2 Extraction procedure set up and validation

6.2.1 The number of components to be analysed shall be chosen so as to measure a significant amount of contaminants that complies with the requirements for a blank (see 6.2.18, NOTE 3).

6.2.2 If the break-in of the component is part of its manufacturing process the extraction procedure should be agreed between parties and included in the inspection document because break-in may alter its initial cleanliness level.

6.2.3 If particles that are detached during transportation of the test component and/or particles from the packaging are to be included in the cleanliness inspection, as agreed upon between parties, they shall be

collected using the appropriate extraction method (e.g. low pressure rinsing). This agreement shall be included in the inspection document.

6.2.4 The effectiveness of the agitation method depends on the following, non-exhaustive list of parameters: type of agitation, duration of agitation, choice of test liquid. A synopsis of the operations to perform is given in Annex A. The detailed description of operating conditions and equipment used in application of this standard to fill, agitate and empty the component constitutes the extraction procedure. This procedure shall be established for each component and reported (for an example of an extraction procedure data sheet see Annex B).

6.2.5 If needed for reporting results and if not specified, determine the controlled volume and/or controlled surface area of the component under examination (see Annex B of ISO 16232-10:2007). Report and/or specify their values in the inspection document.

6.2.6 Before starting to set up or validate any extraction protocol/equipment, it is necessary to perform an initial blank test to know the cleanliness of the equipment. This is performed after cleaning the equipment and the initial blank shall exhibit values stated in 6.3.3.

NOTE Conditioning and cleaning serves the purpose of obtaining a suitable cleanliness level of the inspection set-up. It is recommended that a basic procedure for conditioning the inspection set-up be defined. For example, by performing a cleanliness analysis of a defined volume of liquid after the cleaning procedure of the set-up, it can be determined whether the inspection environment is suitable for carrying out a validation procedure.

6.2.7 If necessary, demagnetise the component and/or clean those external surfaces which are not involved in the cleanliness test.

The external surface should be cleaned in a physically different place from where extraction is to be carried out. Ensure that no particles are deposited on or removed from controlled surfaces. For example, if the component is of large size (like a tank), clean only those external surfaces which might contribute to contamination during the extraction process.

6.2.8 If necessary, remove covers and other plugs fitted for transport of the component. If the component contains a shipment liquid, empty it out, measure its volume and analyse the particles according to Clause 7.

NOTE Removal of plugs might generate particles that contribute to the original contamination.

6.2.9 If dismantling is necessary to obtain access to all the surfaces to be inspected, do so with care.

NOTE Any operation of dismantling might generate particles which could be added to or lost from the original amount of particles.

6.2.10 Transfer a quantity of test liquid whose volume is known to within $\pm$ 5 % and is between 30 % and 40 % of the total volume of the component into a clean sample container, such as a graduated measuring cylinder or graduated beaker.

NOTE This volume may be calculated from the mass of test liquid used.

6.2.11 Carefully pour the volume of test liquid measured in 6.2.10 into the component, then reseal the component so that no contamination is introduced.

6.2.12 Shake the component vigorously in all directions appropriate to ensure thorough agitation of the extraction liquid within all the hollow parts. The agitation protocol shall be adapted to the geometry, dimensions and mass of the component and shall be detailed in the Inspection Document.

For example, a typical protocol for a brake fluid tank is to agitate at 100 to 200 cycles per minute, with an amplitude of 50 to 150 mm, and for a duration of 5 to 15 seconds.

6.2.13 Empty the component:

— by gravity into either :

  — the funnel of the vacuum filtration equipment directly, or

  — a clean collecting equipment (5.6),

— or by using the vacuum suction system (5.5).

Ensure:

— that all of the liquid introduced in 6.2.11 and the suspended particles are recovered during the emptying process;

— that the liquid does not come into contact with any controlled surface not subject to the test.

6.2.14 When necessary, transfer all of the extraction liquid to the analysis equipment by means of a clean sample container(s).

NOTE Depending upon the concentration of particles observed in the extraction liquid, it may be necessary to divide the total volume among several sample containers to facilitate their subsequent analysis, to avoid:

— either clogging of the membrane filter during filtration;

— the saturation of an APC or ;

— overlapping particles in the case of microscopic analysis.

Where appropriate, the inner surface of the test component container shall be rinsed with clean test liquid in order to collect any particles detached during transportation of the test component.

6.2.15 Carefully rinse the collection equipment (see 5.6).

6.2.16 Analyse the extraction liquid in accordance with Clause 7 and label the result obtained as S_1.

6.2.17 Repeat 6.2.10 to 6.2.16 twice more on the same component, using, when necessary, a different container for each extraction liquid sample and label the result obtained as S_2 and S_3.

NOTE The extractions should be made directly one after the other.

6.2.18 Validate the contamination extraction procedure to ensure its efficacy as follows:

a) for each of the three samples analysed in 6.2.16 and 6.2.17, establish the total mass of contaminants and/or the total number of particles. For the particle count, this is applicable to the total number of particles larger than the smallest particle size specified in the inspection document. This particle size shall be chosen to enable counting significant numbers of particles;

b) divide the result of the last sample by the sum of all the values obtained in 6.2.18 a);

c) if the value obtained is less than or equal to 10 %, the end-point is reached and the extraction is completed.

NOTE 1 This procedure enables the extraction curve to be drawn and the end-point ($\leqslant$ 0,10) to be demonstrated (see Figure 1).

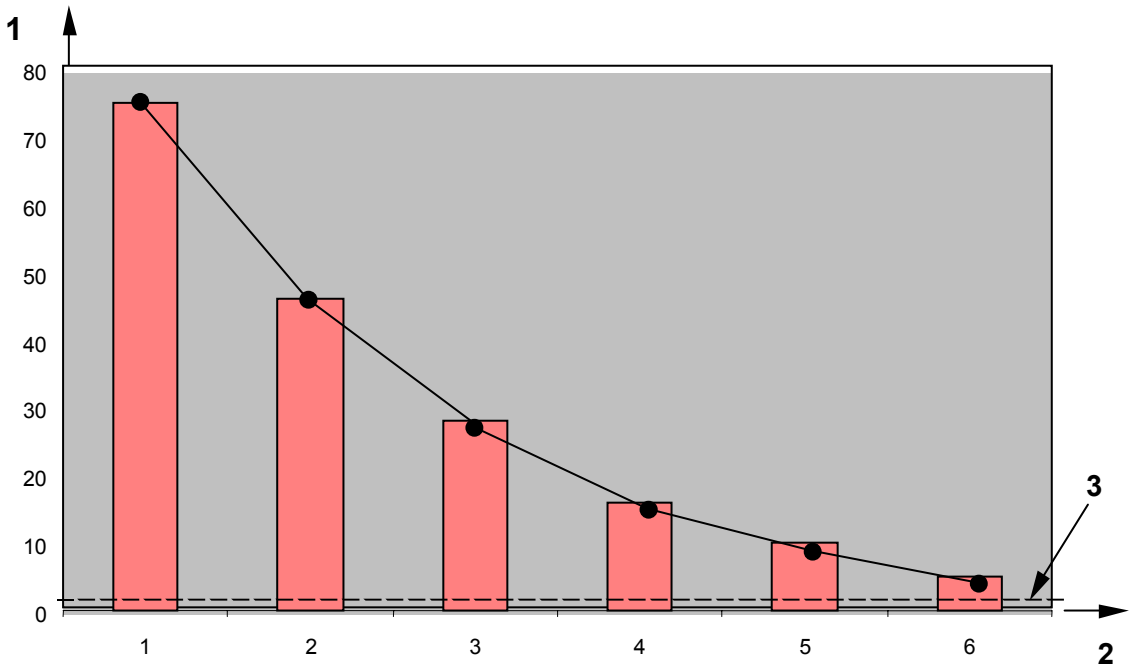
NOTE 2 The cleanliness level of the component is the sum of the extractions.

NOTE 3 In some cases (for example: a very low contamination level, difficulties in extracting particles, inappropriate blank level, etc.) the extraction curve may not be of the form seen in Figure 1. If this is the case, it is ensured that all extraction parameters have been properly investigated.

d) If the value obtained is $> 0{,}10$, a further extraction is necessary. Repeat stages 6.2.10 to 6.2.16 until the last sample S_n produces a result $\leqslant 0{,}10$ of the total of the whole samples ($S_n \leqslant \frac{10}{100}\sum_{i=1}^{n} S_i$).

6.2.19 If six extractions have been performed without reaching a value $\leqslant 0{,}10$, then the extraction parameters are not suitable and shall be modified. Repeat operations 6.2.10 to 6.2.18 with new parameters on a new component.

6.2.20 If this criterion is not fulfilled, set up a new extraction protocol and validate it according to 6.2, or apply another extraction method as defined in ISO 16232-3, ISO 16232-4 or ISO 16232-5.



Key

1 cleanliness level of S_i

2 extraction samples, i

3 blank level

Figure 1 — Example of extraction curve

6.3 Blank test

6.3.1 Sources of blank contamination

6.3.1.1 The blank value accounts for contamination resulting from handling and testing the component, beginning when it is unpacked and ending after the analysis procedure. Main sources of blank contamination are:

— environment (air, operator, working area, etc.);

— test liquid;

— all non-component surfaces that come into contact with the extraction liquid such as containers and equipment for collecting and sampling the extraction liquid;

— analysis of the extraction liquid;

— membrane filter or optical particle counter and associated equipment;

— handling during preparation and analysis of extraction liquid samples.

The blank value results from the combination and interaction of the above factors being applied for a specific test task.

6.3.1.2 The cleanliness of the environment where the cleanliness inspection is performed should be known and be consistent with the presumed cleanliness of the component to be tested. This is validated when performing the blank test.

6.3.1.3 If the blank value level shifts towards higher values, the sources of blank contamination shall be investigated in order to avoid the specified blank level being exceeded in the future.

6.3.2 System blank test

6.3.2.1 A blank test is performed to verify that the operating conditions, equipment and products used in the extraction procedure do not contribute a significant amount of contamination to the component analysed. To ensure process consistency, a blank test should be performed at regular intervals using identical test parameters.

6.3.2.2 For the determination of system blank values, identical conditions as the one applied during testing of the component shall be applied but with the component omitted.

The blank value shall be determined and shall comply with the requirements for each analysis method specified in the inspection document.

6.3.2.3 Proceed as specified in 6.2.10 to 6.2.16 with the same equipment and total volume of test liquid as required for analysis of the component, but without the component.

6.3.2.4 Analyse the extraction liquid as specified in Clause 7.

6.3.3 Blank value

6.3.3.1 General

The acceptable blank value depends on the presumed or specified cleanliness level of the component(s) and depending on the analysis method, is as follows.

6.3.3.2 Gravimetric analysis

Less than 10 % of the presumed or specified gravimetric cleanliness level.

NOTE Using a 4-digit balance under uncontrolled environmental conditions (uncontrolled humidity and temperature) the minimum measurable blank value is 0,3 mg. Thus at least 3 mg should be collected during the component test in order to meet the 10 % criterion.

6.3.3.3 Particle counting and sizing

a) Particle counts: less than 10 % of the presumed or specified numbers, at the relevant sizes, each calculated number being rounded down.

EXAMPLE:

For one particle size, the specified number is 16,
$16 \times 10\ \% = 1{,}6$
rounded value = 1
Conclusion : 1 particle is accepted in the blank.

NOTE The sizes specified in the inspection document for the blank should be as close as possible to the maximum particle size acceptable for the component and chosen to allow counting significant numbers of particles.

b) Maximum particle size: no particle at the ISO 16232-10 size range next lower to half of the presumed or specified maximum particle size.

EXAMPLE:

Maximum acceptable particle size X = 350 µm → 350 µm/2 = 175 µm.

This is size class G according to ISO 16232-10.

Next lower size class is F. That means no particles larger than 100 µm for the blank.

c) If the component cleanliness level is neither presumed nor specified, the blank shall exhibit:

— less than 4 000 particles larger than 5 µm and less than 500 particles larger than 15 µm per 100 mL of extraction liquid;

— no particle larger than 50 µm.

6.3.3.4 If the blank level exceeds 10 %, it is possible to increase the number of test components analyzed in order to collect more particles and thus fulfill the 10 % limit.

6.4 Component routine test

6.4.1 A synopsis of the procedure is given in Annex C.

6.4.2 Apply the extraction procedure described in 6.2.1 to 6.2.15 to the component as many times as determined by the validation procedure and analyze the whole extraction liquid volume as specified in Clause 7.

If the particles that are detached during transportation of the test component and/or particles from the packaging are to be included in the cleanliness inspection, as agreed upon between parties, they shall be collected using the appropriate extraction method (e.g. low pressure rinsing). This agreement shall be included in the inspection document.

NOTE It may be possible to use a combined extraction method by using longer time than that used to validate the extraction procedure provided a sufficient agitation is possible. In this case, the resulting cleanliness level may differ. This simplified method should be validated and should be agreed between parties and included in the inspection document.

6.4.3 When several identical components are measured by a validated method, the cleanliness level of each extraction liquid sample is not required to be measured. All the liquids collected can then be mixed and analysed as specified in Clause 7

NOTE When the extraction method is applied to several components due to their high level of cleanliness (in relation to the value of the blank level, etc.), it is not necessary to measure the contamination level of each extraction sample. All the liquids collected are then mixed and analysed as specified in Clause 7.

7 Analysis of the extraction liquid

7.1 All of the extraction liquid shall be analysed by the method appropriate to the expression of the result of the cleanliness inspection as specified in the inspection document:

— gravimetric analysis in accordance with ISO 16232-6;

— particle sizing and counting by microscopic analysis in accordance with ISO 16232-7;

— particle nature analysis by scanning electron microscopy and EDX in accordance with ISO 16232-8;

— particle sizing and counting by light extinction automatic particle counter in accordance with ISO 16232-9.

7.2 The analysis shall relate to the total volume of liquid used. As specified in the inspection document, all or some of the following liquid samples shall be analysed together provided that the liquids are completely miscible:

— those containing the extracted particles;

— those containing the particles from rinsing the collection equipment;

— those containing the particles from rinsing any packaging;

— those containing all liquid drained from the test item prior to the extraction process.

If the samples contain immiscible liquids then they shall be analysed separately, unless it can be verified that these liquids will not interfere with the analysis method chosen.

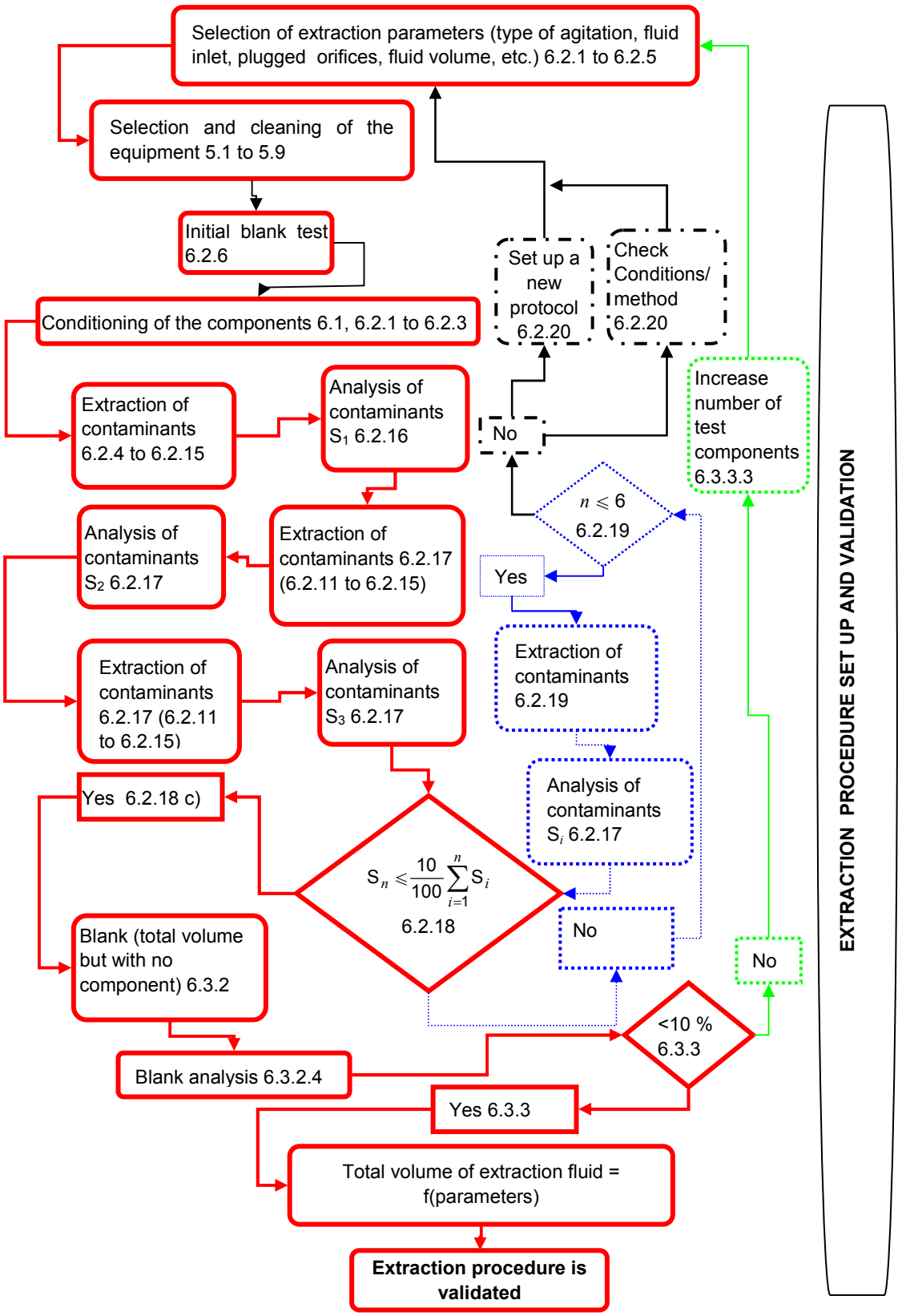
8 Presentation of results

An example of an agitation extraction data sheet is given in Annex B.

Express the results of the cleanliness measurement according to ISO 16232-10.

Annex A
(informative)

Synopsis of the extraction procedure set-up and validation



Annex B
(informative)

Example of data sheet for the extraction procedure by the agitation method

B.1 Operator identification

Date: Operator: Company:

B.2 Test Item Identification

Type:

Reference:

Supplier:

Prior external rinse: ☐ YES ☐ NO

Dismantling: ☐ YES ☐ NO

Demagnetising: ☐ YES ☐ NO

Packaging or container rinsing

☐ YES ☐ NO

Time between production ☐ or shipment ☐ & test

Controlled volume V_C = cm^3

Controlled surface area: A_C = cm^2

Number analysed:

Plugging Caps ☐ YES ☐ NO

Reference :

Analysis of shipment liquid

☐ YES ☐ NO

…….. hours

B.3 Environment

☐ Industrial ☐ Laboratory ☐ Controlled (ISO class 14644-1, class: ----------)

B.4 Test liquid

Identification: _____ Kinematic viscosity: _____mm^2/s Temperature: _____°C

B.5 Conditions of filling/agitation/emptying

Volume of one filling: mL Number of fillings: Openings: see B.2

Means: ☐ Manual ☐ Automatic Ref.

Agitation Duration: sec Frequency: /min Amplitude: /mm

Procedure: see details overleaf and Figure B.7.1 - B.7.4 Emptying: see Figure B.7.1 - B.7.4

B.6 Extraction data and validation

Extraction number (i)	Blank level	1	2	3	4	5	6
Cumulative volume (mL)							
Cumulative mass (mg)							
Cumulative particle count at x µm							
% extraction							

NOTE 1 Extraction is validated when an analysis result is less than or equal to 10 % of the sum of all the results

($S_n \leqslant \frac{10}{100} \sum_{i=1}^{n} S_i$ with $n \leqslant 6$)

NOTE 2 Report particle count data on as many lines as particle sizes used for this validation.

B.7 Illustrations (pictures or drawings to be inserted by the authors of the report)

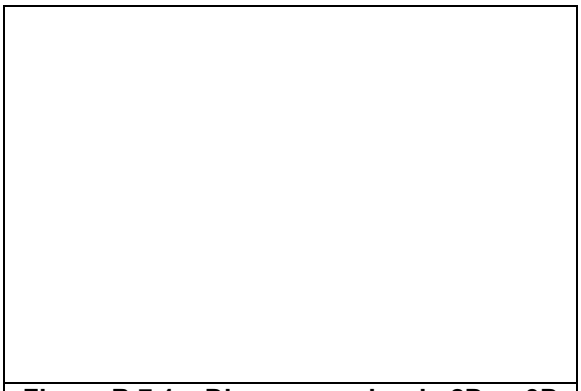


Figure B.7.1 – Diagram or view in 2D or 3D of the component

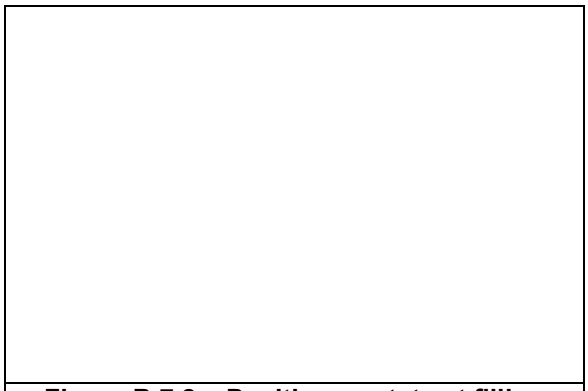


Figure B.7.2 – Position or state at filling (6.2.12)

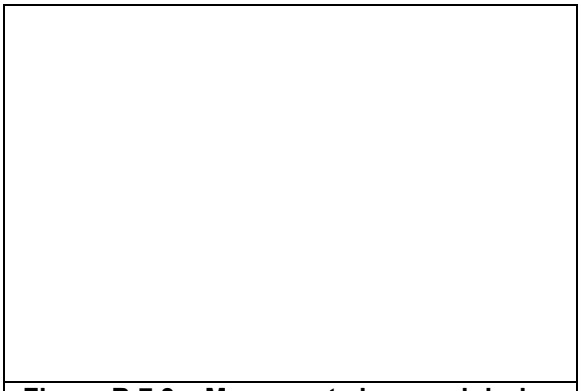


Figure B.7.3 – Movements imposed during agitation (6.2.13)

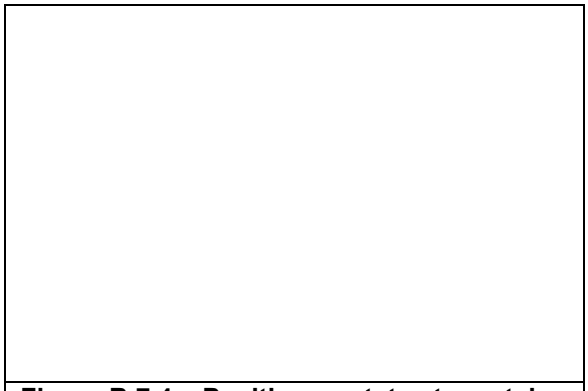


Figure B.7.4 – Position or state at emptying (6.2.14)

B.8 Detailed description of the extraction protocol

(Write a precise sequential list of the functions performed and the conditions of operation of the component before and during the sampling operation.)

Annex C
(informative)

Synopsis of the routine test procedure

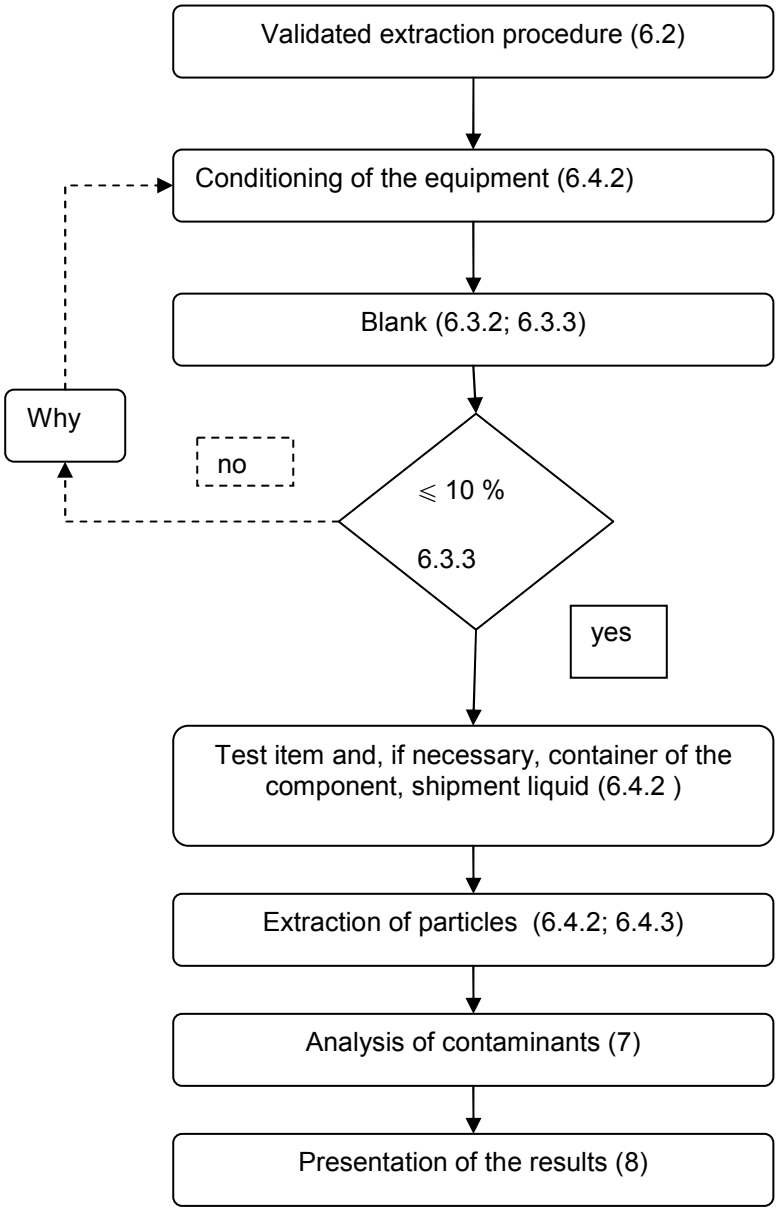


Figure C.1 — Synopsis of the test procedure

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[6] NF E 48-654, *Hydraulic transmissions — Sampling bottle — Dimensions — Cleaning — Conditioning — Identification*

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