BRITISH STANDARD

BS ISO
16232-9:2007

Road vehicles — Cleanliness of components of fluid circuits —

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

ICS 13.040.50; 43.180

British Standards

National foreword

This British Standard was published by BSI. It is the UK implementation of ISO 16232-9:2007.

The UK participation in its preparation was entrusted to Technical Committee MCE/22, Engines for road vehicles.

A list of organizations represented on this committee can be obtained on request to its secretary.

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This British Standard was
published under the authority
of the Standards Policy and
Strategy Committee
on 29 June 2007

ISBN 978 0 580 50920 9

Amendments issued since publication

Amd. No.	Date	Comments

INTERNATIONAL STANDARD

ISO 16232-9

First edition
2007-06-01

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

Reference number
ISO 16232-9:2007(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 16232-9 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.

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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 $\leqslant$ 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.

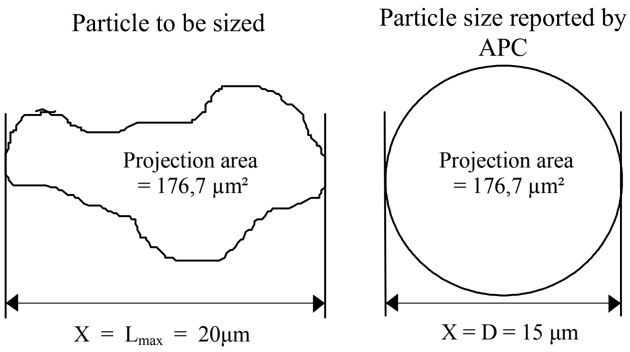


Figure 1 — Particle sizing by APC based on diameter of equivalent projection area

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.

5.7 Health and Safety

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

5.7.2 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.

5.7.3 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.

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

5.7.5 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 apparatus where often volatile liquids are involved.

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

5.7.6 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 and verification

6.1 Verification

An interim check on the calibration status of APC should be made regularly. The performance of the APC can be evaluated by applying samples containing particles of known quantitative size distribution as a reference.

NOTE It may be required to verify that the APC will count zero particles as sizes where such a requirement exists. An example procedure is given in D.3.

6.2 Calibration

The calibration of the APC shall be carried out in accordance with ISO 11171 and ISO 11943 or with other appropriate calibration methods agreed between parties (e.g. using latex spheres).

Calibration of the APC shall be carried out at least every six months, or until enough history with the instrument has been obtained to warrant longer intervals. It shall also be recalibrated when any change is made to the instrument (APC), or its operation becomes suspect.

Flow-meters shall be calibrated for each liquid used for analysis. Calibration of the flow meter shall be carried out in accordance with the manufacturer's recommendation.

7 Procedure

7.1 Summary of the procedure

A summary of a procedure is given in Annex B.

7.2 Validation

The following items shall be checked and established:

a) rinsing procedure for the set-up;

b) attainable blank level for the analysis set-up;

c) time required to ensure that contaminants are put in homogeneous suspension and air bubbles are removed prior to analysis (stabilization period).

An appropriate rinsing procedure for achieving an optimum and stable blank level of the analysis set-up shall be established.

The required cleanliness level of the equipment should be such that contaminants are unable to contribute significantly to the overall result and shall be adapted to the presumed cleanliness level of the component under test. This is validated when performing the blank test of the contaminant extraction method.

A suitable and stable cleanliness level of the set-up which matches the blank test requirements shall be established. The same amount and type of liquid should be used each time the blank test is carried out. For this purpose, a permanent mark indicating the required blank volume of liquid shall be affixed to the outside of the analysis reservoir.

For the determination of the required stabilisation period for a specific liquid, the analysis reservoir should be filled up to its maximum level.

NOTE The performance of the analysis set-up and also the entire analysis procedure can be evaluated by applying sample containing particles of known size distribution as a reference.

7.3 Conditioning of the analysis set-up

The reservoir, sensor and associated pipework shall be cleaned prior to use by rinsing with filtered test liquid. This can be achieved by filling the reservoir to its highest level with filtered liquid and rinsing through at a flow rate higher than the analysis flow rate.

If the system has previously been used to analyse a liquid which is not miscible with the test liquid to be analysed, the analysis set-up will need special cleaning (for guidance see Annex D).

After rinsing the set-up, partially drain the reservoir to a minimum volume (for example, 10 mL to 50 mL). The minimum volume should be chosen such that no air is introduced into the sampling line of the sensor during the process of emptying the reservoir.

7.4 Blank check of the analysis set-up

Verify the actual blank level of the analysis set-up by analysing a defined volume of filtered test liquid. The cleanliness level is acceptable when the result of the blank check is in compliance with the base-level established during the validation of the set-up.

Ensure that the sensor of the APC has been switched on for long enough to become stabilised.

The selection of the size intervals of the APC shall be in accordance with 5.5.3.

At the beginning of the following procedure, make certain that the reservoir contains the minimum volume of filtered test rinsing liquid to ensure that no gas or air is introduced into the supply line of the APC.

a) Gently fill the reservoir with compatible test liquid up to the blank volume mark.

b) For stabilisation, gently stir the contents of the reservoir for a minimum period of time as determined by the validation procedure of the set-up.

NOTE 1 A stirrer speed that is too high can introduce bubbles to the liquid which will cause erratic counting and hence errors.

c) Afterwards, set the APC to acquire data and to count particles and enable the test liquid contained in the reservoir to be fed through the sensor. The test liquid will now be analysed. If the counting mode of the APC is set to “time”, set the delay time of the APC to zero in order not to miss any particle passing through the sensor.

NOTE 2 Depending on the set-up configuration, it may be necessary to adjust the working flow rate during analysis.

d) When the test liquid volume within the reservoir falls to the minimum established in 7.2, stop the data acquisition of the APC and stop the flow through the sensor.

e) Repeat stages a) to d) and accumulate the data to determine the system blank level.

Check whether the blank level is in compliance with the required blank level for the system (see 7.2). If the blank value is not satisfactory:

- check set-up and conditions;
- repeat previous steps.

7.5 Preparation of the extraction sample

7.5.1 Pre-assessment – suitability and dilution

Remove any visible contamination from the exterior of the sampling container(s) using a wetted lint-free cloth.

Liquid samples which are found on visual examination to contain contamination which is likely to affect the performance of the APC shall not be counted by this method.

If the presence of immiscible liquids is seen in the sample, do not analyse the sample by this method, because the immiscible liquid will interfere with operation of the instrument. The sample shall be analysed using the optical microscope in accordance with ISO 16232-7.

The sample should be analysed in an undiluted form. However, dilution may be necessary to reduce the density of the particle population. Guidance for dilution is given in Annex D.

a) Check if dilution is necessary as follows:

   Calculate the total number of particles according to the cleanliness specification of the component under test and divide it by the total volume of the extraction sample. If the resulting concentration exceeds the limit of 80 % of the coincidence-limit, dilution of the sample is necessary.

b) If the concentration of the liquid sample can not be calculated it is necessary to analyse an exploratory sample:

   Take a defined portion of the extraction sample. Dilute the portion by 1:10. Condition and analyse the diluted portion as described for test samples within this document. The result of this exploratory analysis indicates whether the sample can be analysed directly or what the optimum dilution ratio will be for the remaining extraction sample. The final dilution ratio shall be selected to give a particle count of less than 80 % of the stated coincidence. For the final reporting of the particulate count of the extraction sample it is important also to include the total number of counts obtained from the exploratory sample.

7.5.2 Re-dispersion of contaminants

If the sample is left to stand for a period of time, agglomeration of contaminants will occur. The agglomerates shall be broken up to disperse the particles evenly. This may be done either by manual or automatic shaking or by using an ultrasonic bath. The method chosen shall not alter the original particle size distribution.

7.6 Sample analysis

Conditioning and preparatory work are necessary in order to avoid erroneous results, which can be caused by liquid-borne gas bubbles.

The sample shall be analysed immediately after re-dispersion and degassing.

Ensure that the sensor of the APC has been switched on for long enough to become stabilised (detection optics and electronics).

The setting of the size intervals of the APC shall be according to those stated in the Inspection Document and consistent with the requirements specified in ISO 16232-10.

At the beginning of the following procedure, make certain that the reservoir contains the minimum volume of filtered test liquid to ensure that no gas or air is introduced into the supply line of the APC.

a) Gently empty the entire liquid sample into the reservoir and start the stirrer.

NOTE 1 A stirrer speed that is too high can introduce air bubbles to the liquid.

b) Rinse the inner sides of the sample container with pre-filtered test liquid and gently empty the extraction liquid into the reservoir.

c) For stabilisation, gently stir the contents of the reservoir for a minimum period of time as determined by the validation procedure.

d) Set the APC to acquire data and enable the liquid contained in the reservoir to be fed through the sensor and immediately count particles . Analyse the liquid. If the counting mode of the APC is set to time set the delay time of the APC to zero in order not to miss any particle passing through the sensor.

NOTE 2 Depending on the equipment configuration, it may be necessary to adjust the working flow rate during analysis.

e) When the liquid volume within the reservoir falls to the minimum established in 7.2, stop the flow through the sensor and stop the data acquisition of the APC.

f) Repeat steps a) to e) until all the sample containers have been analysed.

g) After all extraction samples have been analyzed, remove residual particles by rinsing the surfaces of the reservoir using liquid from a pressurized dispenser. Start at the uppermost surface and gradually work down to the bottom. Repeat stages c) to e) and note the particle counts.

NOTE 3 For the rinsing process to be most effective, a fan shaped nozzle has proved to be the most effective.

Repeat step g) until either the “end-point” is reached, i.e. where the numbers of particles extracted in this rinse is $<$ 10 % of the total extracted, or the blank value is reached.

The total data acquired from the samples by the APC during the analysis procedure stated above represent the particle count of the liquid sample under test.

8 Results

8.1 Calculations

To obtain the total number of particles per test component, the particle counts from the individual analysis steps are summed, i.e:

a) the total number of particles recorded in the analysis of any shipping liquid drained from the test component prior to extraction if requested in the inspection document;

b) the total number of particles at each size recorded for any exploratory analysis, e.g. optimization of dilution ratio [7.5.1 b)];

c) the total number of particles recorded for each extraction sample analyzed [7.6 f)];

d) the total number of particles at each size rinsed from the test equipment after the analysis of the extraction samples [7.6 g)].

A typical test report is shown in Annex E.

8.2 Expression of results

Express the results of the component cleanliness examination in accordance with ISO 16232-10.

Annex A
(informative)

APC analysis

This part of ISO 16232 does not cover the use of APC for on-line analysis of the liquid in the fluid lines. Nevertheless liquids from sampling lines may be collected in clean sample containers and analysed as described in this part of ISO 16232.

Only clear, single phase liquids that are devoid of optical interfaces may be analysed with APC, according to the instrument specification.

Due to the specific calibration and detection characteristics of APC, the results of APC analysis are not numerically comparable to results obtained by other methods, such as microscopy.

The reported size of the particles detected by APC corresponds to the diameter of a sphere having the same projected area as the particles analysed, as shown in Figure 1. APC is not capable of detecting the maximum dimension of irregularly shaped particles.

Annex B
(informative)

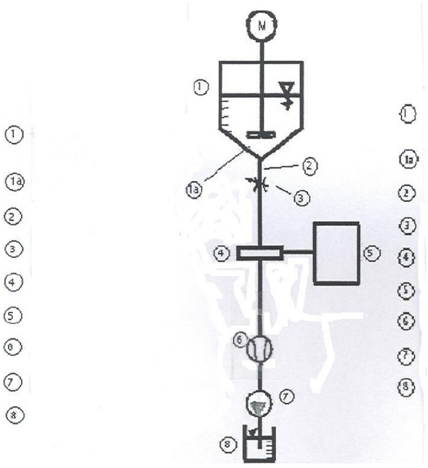
Summary of the analysis procedure

B.1 Summary of the analysis procedure

No	Description	Action	Comment	Frequency
1	Conditioning of the equipment	Cleaning, rinsing and stabilising of the analysis set-up in order to: - displace any inclusions of air bubbles - displace any incompatible liquids - disqualify unsuitable sample for analysis - provide suitable blank level	Liquid used shall be compatible with the test sample to be analysed	Prior to each analysis
2	Measuring of the blank level of the analysis set-up	Preparation and analysis of a representative blank sample	Amount of the volume be chosen according to good practice	Regularly
2a	Preparation of the system blank sample	sampling stabilising	—	Each analysis
2b	Measuring of the system blank sample	Sizing and counting of the particles by using APC	—	Each analysis
2c	Assessment of the system blank level	Check if the blank level is in compliance with the base level established for the system.	If the blank value is not suitable: check set-up and conditions repeat previous steps	
3	Characterisation of the test sample	Preparation and analysis of the extraction liquid	—	Each analysis
3a	Preparation of the test sample	- Dilute if necessary - Re-dispersion Degassing - Stabilising	Check whether dilution is necessary or not. It may be necessary to carry out an exploratory sample.	Each analysis
3b	Analysis of the test sample	Sizing and counting of the particles by using APC	Repeat steps 3a to3e in case the extraction sample is divided into several sample containers	Each analysis
3c	Analysis of the final rinsing sample	Sizing and counting of the particles by using APC	—	Each analysis
4	Reporting and expression of results	The result of particle measurement shall be related to the component from which the sample originally was extracted.	Totalise the counts determined for the relevant samples	Each analysis

Annex C
(informative)

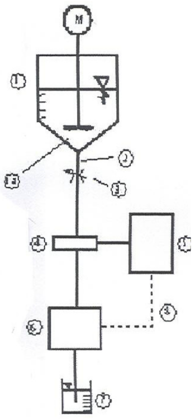
Example of configuration - Sampling apparatus



Key

1	lockable graduated reservoir and stirrer
2	flexible tube
3	clamp valve
4	APC sensor
5	APC instrument
6	flow meter
7	adjustable suction pump
8	graduated drain

Figure C.1 — Continuous flow configuration based on a non-pulsating suction pump



Key

1	lockable graduated reservoir and stirrer
2	flexible tube
3	clamp valve
4	APC sensor
5	APC instrument
6	syringe sampler
7	graduated drain
8	remote line

Figure C.2 — Non-continuous flow configuration based on an automatic syringe sampler

NOTE 1 It is advisable that the outlet of the reservoir be at the bottom of the conical section of the reservoir and that the flow be vertically downwards so that particles cannot settle in the pipes. It is advisable that changes in area be eliminated.

NOTE 2 If a gas is used to force the liquid through the sensor, the gas should be filtered through a suitable filter and should be free from oil and water.

Annex D
(informative)

Supplementary procedures

D.1 Sample dilution

D.1.1 General

Sample dilution may be necessary to reduce the particle concentration so that it is below the coincidence limit of the instrument.

The process of dilution may introduce errors in particle count data because of the addition of extraneous contaminant, which will be magnified by the dilution ratio.

To reduce such errors, dilution should be performed under clean conditions, and all equipment used in the procedure should be thoroughly cleaned using approved procedures.

NOTE By diluting, the refractive index of the sample may be changed, and this may affect particle characterisation.

D.1.2 Single-step dilution

Calculate the dilution ratio required.

Verify the cleanliness level of the diluent before use.

Use an empty pre-cleaned sample bottle (= diluent bottle) which is large enough to contain the diluted sample.

Add a suitable volume of filtered diluent liquid by using the mass or volumetric method.

Redisperse the contents of the test sample bottle and transfer it completely into the diluent bottle

a) Rinse the inner surface of the test sample bottle with filtered diluent liquid, cap the bottle and shake it vigorously for 5 s.

b) Empty the content of the bottle into the diluent bottle.

The content of the dilution sample bottle is now prepared for carrying out the analysis procedure for extraction samples.

NOTE ISO 11500 is used for mass and volumetric dilution methods.

D.2 Analysis of different liquids

Care should be taken when the analysis set-up is used to analyse non-compatible liquids.

Films or droplets of the previous liquid may remain in the set-up, and this will lead to erratic counting. The procedure for changing liquids involves flushing with a succession of solvents, each one compatible with the preceding one. For example, to change from aqueous solution to an hydrocarbon based test liquid, a typical sequence is:

a) flush with distilled or de-ionised water;

b) flush with propan-2-ol;

c) flush with petroleum spirit or a suitable solvent with similar properties;

d) flush with solvent compatible with the solvent contained in the analysis sample.

D.3 Checking the zero count-rate of the instrument

Use a clean liquid that is drawn through the APC. Close any valves located downstream and/or upstream the sensor such that no liquid can flow through the sensor. Eventually disconnect and/or disable any remote-controlled sampling apparatus. Set the APC to counting mode. If no significant counts occur the zero count-rate is verified.

NOTE This procedure is only suitable for APC that do not get a flow reading error when the flow is disabled.

Annex E
(informative)

Example of standard analysis data sheet particle sizing and counting in liquid samples by automatic light extinction particle counter

E.1 Analysis protocol

In addition to the analysis report as described in ISO 16232-10, the following data should be recorded:

Data-sheet for particle analysis of extraction samples by using APC			
Analysis parameters		Technical Data	
1	Number of extraction sample containers:	1	Type and manufacturer of APC:
2	Total original volume of the extraction sample:	2	Calibration method (please underline): Latex / ISO-MTD:
3	Test liquid:	3	Dynamic size range of the instrument:
	Dilution liquid:	4	Coincidence level:
4	Rinsing liquid:	5	Type and manufacturer of sampling apparatus:
5	Sampling method:		
6	APC flow rate:		
7	Extraction liquid temperature:		
8	System blank level found suitable (please underline): YES / NO		
9	Concentration during final analysis found below coincidence concentration (please underline): YES / NO		

E.2 Analysis results

Example for the reporting based on differential data:

	Size class	B	C	D	E	F	G	H	I
	Size [µm] (ISO 16232-10)	$5 \leqslant X <$ 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
	Size [µm(c)] (ISO 11171)	$4 \leqslant X <$ 14	$14 \leqslant X <$ 21	$21 \leqslant X <$ 38	$38 \leqslant X <$ 70	-----------	----------	----------	----------
Blank value									
	Exploratory sample (optional)								
	Extraction sample(s)		43	31	6	4	1	2	0
	Drained liquids								
	Final rinsing sample		12	9	5	2	0	1	0
Total number			55	40	11	6	1	3	0

NOTE 1 Due to the specific calibration and detection characteristics of APC, the results of APC analysis are not numerically comparable to results obtained by other methods, such as microscopy.

NOTE 2 The particles cannot be measured in the size range $>$ 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.

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