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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Aerospace — Fluid systems and components — Methods for system sampling and measuring the solid particle contamination of hydraulic fluids

Aéronautique et espace — Systèmes de fluides et éléments constitutifs — Méthodes de prélèvement et de mesure de la contamination particulaire solide des fluides hydrauliques

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Aerospace — Fluid systems and components — Methods for system sampling and measuring the solid particle contamination of hydraulic fluids

0 Introduction

0.1 General

The design of modern hydraulic equipment for aerospace purposes, its use and performance are widely determined by the type and condition of the applicable hydraulic fluids.

The quality and serviceability of these hydraulic fluids are dependent on various factors (e.g. thermal stability, viscosity), in particular on the level of solid particle contamination which requires regular control if it is to be kept within the given limits.

To obtain comparable test results, uniform test methods as given in this International Standard have to be used.

As a result of the rapid development and improvement of hydraulic systems and their components, which have to meet critical requirements, the problem of solid particle contamination of hydraulic fluids has steadily increased. The need for maintaining a specified standard of fluid cleanliness in hydraulic systems requires continuous control of the number and size of the solid particle contaminants.

0.2 Solid particle contamination

Solid particle contaminants can be the cause of abrasion and wearing, thereby shortening the life of the components in a hydraulic system.

In a hydraulic system

a) components are subject to erosion (primarily in components with higher fluid velocities);

b) all moving parts are subject to wear by abrasion;

c) control valves are subject to silting (settlement of fine particles on the control bore).

0.3 Causes of solid particle contamination

Solid particle contamination of hydraulic fluids may be system-generated, introduced from the outside, in-built or maintenance-generated, e.g.

a) dust particles in the air;

b) metal particles, produced during the manufacture of parts;

c) sand residues on castings;

d) abrasion of seals;

e) oxide layers on welding seams and on heat-formed or heat-treated steel parts;

f) chemical and physical changes in the condition of hydraulic fluids;

g) maintenance of hydraulic systems (e.g. fibres, secondary contamination, etc.);

h) wear of components;

i) ingress of particles via piston gland seals.

0.4 Layout of this International Standard

This International Standard is sub-divided into the following sections:

— Section one: Cleaning of apparatus and sample bottles (clauses 3 to 7);

— Section two: Sampling (clauses 8 to 11);

— Section three: Methods for determining solid particle contamination (clauses 12 to 14);

— Section four: Test reports (clause 15).

1 Scope and field of application

This International Standard specifies measuring methods for determining the level of solid particle contamination of hydraulic fluids used in hydraulic systems and equipment for aerospace purposes. Three methods are specified in this International Standard:

a) microscopic method;

b) automatic particle counting method;

c) gravimetric method.

In addition, this International Standard lays down the procedures for cleaning the apparatus and sample bottles, and specifies the solvents to be applied and how these solvents shall be prepared. Requirements as to the selection of the sampling point and to different sampling methods are also specified.

2 References

ISO 3722, *Hydraulic fluid power — Fluid sample containers — Qualifying and controlling cleaning methods.*

ISO 4402, *Hydraulic fluid power — Calibration of liquid automatic particle-count instruments — Method using Air Cleaner Fine Test Dust contaminant.*

ARP 1192 A, *Procedure for calibration and verification.*

Section one: Cleaning of apparatus and sample bottles

3 Cleanliness

When establishing the solid particle contamination of hydraulic fluids, the test results may be adversely affected by not sufficiently taking account of the need for an optimum cleanliness.

In order to obtain meaningful results reproducible at any place and at any time, it is essential to ensure that the preliminary conditions prevent any additional solid particle contamination of the hydraulic fluid sample taken.

Additional solid particle contamination can be caused by using apparatus inadequately cleaned for the measurement.

All apparatus used for determining of the solid particle contamination level of hydraulic fluids shall be thoroughly cleaned before use in accordance with the procedure specified in clause 7. The apparatus shall be resistant to the solvent and the sample fluids.

4 Solvents

The solvents specified in 4.1 to 4.3 shall be used for the cleaning procedure.

4.1 Solvent A: De-mineralized or **distilled** or **de-ionized water,** or **water obtained from the conversion osmosis.**

4.2 Solvent B: 2-propanol (isopropyl alcohol), reagent-pure.

4.3 Solvent C: Petroleum ether, halogenated solvents, or other approved equivalents.

5 Filtration of solvents

5.1 Pressure filtration

5.1.1 Apparatus

5.1.1.1 Pressure tank, stainless steel.

5.1.1.2 Filter-jet spray gun, with filter attachment.

5.1.1.3 Membrane filter, having an aperture size less than or equal to 0,5 µm.

5.1.2 Procedure

The filter-jet spray gun (5.1.1.2), with a membrane filter (5.1.1.3) fitted, shall be connected by a hose to the pressure tank (5.1.1.1) containing the solvent. The gun shall produce a concentrated jet of filtered solvent for cleaning the surfaces. Precleaning is not required.

5.2 Vacuum filtration

5.2.1 Apparatus

5.2.1.1 Vacuum pump.

5.2.1.2 Filtration apparatus, stainless steel or glass.

5.2.1.3 Wash bottles.

5.2.1.4 Membrane filter, having an aperture size less than or equal to 0,5 µm.

5.2.2 Procedure

For vacuum filtration of the solvent, the filtration apparatus (5.2.1.2) shall be fitted with a membrane filter (5.2.1.4) and connected to the vacuum pump (5.2.1.1) by a hose. The solvent shall be filtered through the membrane filter by the vacuum in the flask of the filtration apparatus. The filtrate collected in the flask shall be transferred to the corresponding wash bottle (5.2.1.3).

6 Sample bottles

The sample bottles shall be made from glass, shall have a nominal capacity of 250 ml and a minimum capacity of 150 ml, and shall be sealed by means of caps (preferably of phenolic resin) which will not cause contamination. Otherwise a non-flaking plastic film compatible with the hydraulic fluid shall be used.

7 Cleaning procedures

7.1 General conditions

Staff performing the cleaning procedure shall wear lint-free clothes (e.g. cap, smock frock, boots) to avoid excessive secondary contamination by fibres.

The standard of the workroom shall be such as to ensure that the specified cleanliness standard can be achieved repeatably.

This condition is superfluous when working in a clean environment such as a clean-room or clean-cabinet.

7.2 Cleaning procedure for filtration apparatus and filter-jet spray gun

Before use, every part of the filtration apparatus and filter-jet spray gun shall be cleaned in accordance with the following method:

a) rinse with a degreasing fluid;

b) wash thoroughly in a hot water solution of detergent;

c) rinse twice with hot water (at a temperature of between 40 °C and 60 °C);

d) rinse twice with solvent A (4.1), filtered through a membrane filter having an aperture size less than or equal to 0,5 µm;

e) rinse three times with solvent B (4.2), filtered through a membrane filter having an aperture size less than or equal to 0,5 µm, to remove the water;

f) rinse three times with solvent C (4.3), filtered through a membrane filter having an aperture size less than or equal to 0,5 µm.

In carrying out steps d) to f), care shall be taken to ensure that the whole surface of the apparatus is pressure-rinsed from top to bottom.

7.3 Cleaning procedure for sample bottles

Before use, sample bottles and caps shall be thoroughly cleaned in accordance with the procedure specified in 7.2 a) to f) for filtration apparatus and filter-jet spray gun. In carrying out steps d) to f), care shall be taken to ensure that the whole surface of the sample bottles is pressure-rinsed from top to bottom.

After the last rinse, a small amount of solvent C remains in the sample bottle. The resultant gas pressure inside the sample bottle avoids contamination when the bottle is opened. When using a piece of plastic film, 100 mm × 100 mm in area, the film shall be rinsed with filtered solvent C and then placed over the mouth of the sample bottle with the edges of the film bent downwards and the cap screwed onto the bottle. The cap shall not be tightened too hard to avoid breaking the plastic film.

7.4 Checking and controlling cleaning methods

Checking the cleaned sample bottles shall be performed in accordance with ISO 3722.

Section two : Sampling

8 Sampling — General

When measuring the solid particle contamination of hydraulic fluids, the selection of the sampling point where a sample is taken from the hydraulic system and the way of taking it are of prime significance to the value of the sampling.

9 Apparatus

All apparatus and equipment used in sampling procedures A and B shall be thoroughly cleaned in accordance with the procedure specified in clause 7.

9.1 Sampling procedure A

9.1.1 Sample bottles, as specified in clause 6.

The sample bottles shall not contain more than 200 particles greater than 10 µm nor more than 500 particles greater than 5 µm per 100 ± 5 ml.

9.2 Sampling procedure B

9.2.1 Monitor, containing membrane filters suitable for the microscopic or gravimetric method (see clauses 12 and 14).

9.2.2 Graduated collecting bottle.

10 Sampling point

The sampling point differs from one hydraulic system to another and shall be defined by the system designer. However, the location should be a point where the best chances for a representative distribution of the solid particle contamination are given.

It is advisable, and, in some cases, will be required, to operate the system at full flow to ensure that the sample taken is representative of the aircraft at near flight conditions.

The following criteria shall be taken into account when defining the sampling point:

a) The sampling valve shall be automatic-closing and contain a separate cap with an integral seal capable of withstanding system pressure and be placed at the sampling point without the link tubing being too long.

b) A sample taken from the system reservoir is not necessarily to be regarded as representative for the system.

11 Sampling methods

11.1 Sampling procedure A (using sample bottles)

11.1.1 The sampling valve of the hydraulic system shall be capped. Wipe the cap clean with a lint-free cloth before removing the cap.

Successively open and close the sampling valve three to four times for a short period.

11.1.2 Open the sampling valve and release into a waste container a volume of hydraulic fluid to flush the sampling system (a minimum volume of 100 ml is recommended). In determining the minimum volume to be flushed, full account shall normally be taken of the link pipe volume.

11.1.3 Remove the sealing cap, with plastic film, if used, from the sample bottle (9.1.1) and drain off solvent from the bottle. Fill the sample bottle to 50 % to 70 % (100 ml min.) of its capacity.

11.1.4 Do not actuate the sampling valve throughout the sampling period.

11.1.5 Remove the bottle from sampling stream.

Close the sample bottle with sealing cap, and plastic film, if used, as specified in clause 6.

Close the sampling valve. To avoid contamination do not wipe the sampling valve. Replace the cap on the sampling valve.

11.1.6 Identify the sample as specified in 15.3.

11.2 Sampling procedure B (using the monitor)

11.2.1 Attach the sample-releasing apparatus to the quick disconnect coupling.

Fit the monitor (9.2.1) into the sample-releasing apparatus.

Position the three-way valve so that the hydraulic fluid is diverted by a hose before it reaches the monitor inlet.

11.2.2 Open the sampling valve and release into a waste container a volume of hydraulic fluid to flush the sampling system (a minimum volume of 100 ml is recommended). In determining the minimum volume to be flushed, full account shall normally be taken of the link pipe volume.

11.2.3 Position the three-way valve so that the hydraulic fluid filters through the monitor and flows into the graduated collecting bottle (9.2.2).

11.2.4 Close the sampling valve.

11.2.5 After the desired sample volume (100 ± 5 ml) has been taken, return the three-way valve to its initial position.

11.2.6 Before removing the monitor, attach the injector nozzle and ensure that the remaining fluid passes through the monitor. Then remove the monitor and mount caps.

NOTE — The whole procedure should be carried out with reference to the manufacturer's instructions.

11.2.7 Identify monitor as specified in 15.3.

11.2.8 Transportation of the monitors with the membrane filter from the sampling point to the laboratory shall be carried out with care to prevent contaminants dropping off the membrane. The surface holding the contaminants shall always be directed upwards and determination shall be made as quickly as possible.

Section three: Methods for determining solid particle contamination

Three methods for determining solid particle contamination of hydraulic fluids are laid down in this International Standard:

a) microscopic method (see clause 12);

b) automatic particle counting method (see clause 13);

c) gravimetric method (see clause 14).

12 Microscopic method

12.1 Principle

Filtration of a known volume of fluid under vacuum conditions through a membrane filter with an imprinted grid to collect the solid particle contamination on the filter surface. Counting and grouping of particles into size classes according to their largest dimension.

The filter is rendered transparent for examination under transmitted light and left opaque for examination under incident light. Under incident light, the use of multiple or episcopic illumination is recommended to reduce the core shadows.

12.2 Apparatus

12.2.1 Filtration apparatus, including

— a funnel, glass or stainless steel, approximately 250 ml capacity (150 ml min.);

— a clamping device;

— a glass or stainless steel support, including a sintered glass or stainless steel filter-holder screen.

12.2.2 Funnel cover (e.g. an element of a Petri dish).

12.2.3 Membrane filters, greater than or equal to 47 mm in diameter, with grid, with an aperture size less than or equal to 1,2 µm. Each grid square has 3,08 mm sides corresponding to approximately one-hundredth of the filtering area.

12.2.4 Vacuum flask.

12.2.5 Measuring cylinder, with a capacity of more than 100 ml.

12.2.6 Device for establishing a controllable vacuum.

12.2.7 Filter-jet spray gun, for the distribution of filtered solvent C (a pressure-operated system which discharges the solvent, in the form of a fine jet, through a membrane filter, the aperture size of which is less than or equal to 0,5 µm.)

12.2.8 Forceps, stainless steel, with unserrated tips.

12.2.9 Microscope slides, cover slips having a maximum thickness of 0,25 mm and lower slides with a minimum thickness of 1 mm are recommended, or **Petri dish,** with cover.

12.2.10 Means to obtain transparency of membrane filter.

NOTE — When using immersion oil, the refractive index shall be comparable to that of the membrane filter.

12.2.11 Monocular, binocular or projection microscope, for magnification X40 and X100 for incident light and, in addition, X400 for transmitted light, equipped with micrometer filar eyepiece for adjustment to the magnifications specified.

12.2.12 Stage micrometer, graduated in divisions of 0,1 mm and 0,01 mm.

12.2.13 Microscope light, variable intensity, used as source for incident or transmitted light.

NOTE — Prior to use, filtration apparatus (12.2.1), funnel cover (12.2.2), forceps (12.2.8), Petri dish and microscope slides (12.2.9) shall be cleaned in accordance with the procedure specified in 7.2.

12.3 Procedure

The use of a dust-free chamber with laminar flow for the filtration serves to reduce extraneous contamination.

12.3.1 Blank test filtration

This procedure shall be carried out periodically in accordance with quality control requirements to check the validity of the cleaning and test procedures.

12.3.1.1 Using forceps, remove a membrane filter (12.2.3) from the storage container and rinse it on both sides with filtered solvent C.

12.3.1.2 Place the membrane filter on the support of the filtration apparatus, grid face upwards, lower the previously cleaned funnel without pushing it over the filter and secure the assembly with the clamping device.

12.3.1.3 Pour 100 ml of filtered solvent C into a sampling bottle, cleaned in accordance with 7.3, and shake thoroughly.

12.3.1.4 Remove funnel cover, pour the contents of the bottle into the funnel and replace cover.

12.3.1.5 Apply the vacuum device (12.2.6) slowly up to 87 kPa (0,87 bar) max. and filter until the liquid level in the funnel is approximately 10 mm.

12.3.1.6 Remove the cover, rinse the funnel side walls with filtered solvent C and take care to direct the jet of the spray gun (12.2.7) onto the funnel walls to avoid contact with the liquid and to obtain a better particle distribution on the filter. Replace cover.

12.3.1.7 After evacuation of the solvent, remove cover, clamping device and funnel.

12.3.1.8 Turn off the vacuum.

12.3.1.9 Remove the filter and prepare it for the chosen method of microscopic examination as follows:

Under incident light

Place the filter in a cleaned Petri dish or between two cleaned microscope slides to read off under incident light.

Under transmitted light

Render the filter transparent (12.2.10) on a cleaned microscope slide.

In both cases, the membrane filter shall be totally flat, grid face upwards, and the grid lines shall be parallel to the slide edges.

12.3.1.10 Count the contaminant as specified in 12.5.

12.3.1.11 The blank count shall not exceed 10 % (rounded) of the total allowable sample count. If the blank analysis exceeds this value, the apparatus shall be recleaned (as specified in 7.2) and the procedure shall be repeated.

12.3.2 Sample filtration

12.3.2.1 Follow the same procedure as specified in 12.3.1.1 and 12.3.1.2.

12.3.2.2 Shake the sample bottle, manually or mechanically, until the contaminants are homogeneously suspended and remove the sample bottle cap.

12.3.2.3 Pour off 100 ± 5 ml of the sample into the measuring cylinder (12.2.5).

12.3.2.4 Pour the sample into the funnel and replace the cover.

12.3.2.5 Follow the same procedure as specified in 12.3.1.5 to 12.3.1.10.

12.4 Microscope calibration

This procedure shall be carried out periodically in accordance with quality control requirements.

12.4.1 Place a stage micrometer (12.2.12) under the microscope (12.2.11), fitted with suitable eyepieces and objectives.

12.4.2 Adjust the objectives and focus eyepieces on to the graticule of the stage micrometer.

12.4.3 Calibrate the filar micrometer in each eyepiece by determining the number of divisions of the stage micrometer which corresponds to the entire length of the scale of the filar micrometer. This method gives the micrometre value of a division of the filar micrometer for each objective used.

12.5 Sample counting

The use of a dust-free chamber with laminar flow for counting serves to reduce extraneous contamination.

12.5.1 Preparation

Place the microscope slide or the Petri dish with the membrane filter on the microscope stage and adjust the focus and grid orientation.

12.5.2 Particle size ranges and selection of appropriate magnification

The particles, p, shall be classified according to their largest dimension in the following size ranges:

- 5 µm $< p \leqslant$ 15 µm
- 15 µm $< p \leqslant$ 25 µm
- 25 µm $< p \leqslant$ 50 µm
- 50 µm $< p \leqslant$ 100 µm
- 100 µm $< p$
- Fibres

NOTES

1 A fibre is defined as a particle greater than 100 µm the length of which exceeds the width by at least 10 times.

2 Classification of particles according to size ranges selected by the user is allowed.

Select the magnification appropriate to the particle size ranges to be counted and to the method to be used from table 1.

Table 1 — Particle size ranges and magnifications for sample count

Particle (p) size range µm	Magnification	
	under incident light	under transmitted light
$5 < p \leqslant 15$	X100	X400
$15 < p \leqslant 25$	X100	X100
$25 < p \leqslant 50$	X100	X100
$50 < p \leqslant 100$	X40	X40
$100 < p$	X40	X40
Fibres	X40	X40

12.5.3 Particle distribution

Examine the total filtering area for good particle distribution. If the distribution is not uniform, the statistical counting procedure cannot be used and, consequently, either the liquid sample should be re-run or a total particle count performed.

12.5.4 Statistical counting procedure

The counting procedure shall be applied to a number of finite areas, depending on the magnification used, size range concerned and level of contamination of the sample.

Carry out the counting on a certain number of unit areas and the statistical result is calculated by multiplying by the ratio of the total filtration area to the evaluated area (sum of the unit areas).

NOTE — The unit area is defined by the operator, it may be, for example,

- the area of a filter grid square, excluding grids bounded by the filter circumference;
- the area scanned by the micrometer between two reference points.

It should be of such an area that not more than 50 particles of the size to be counted are contained within it.

For particles > 50 µm, count the total filtration area.

For particles ⩽ 50 µm, determine the number of particles within a given size range by counting these particles on a limited number of squares and by transferring this result to the effective filtration area (100 squares). If this estimation yields

- fewer than 50 particles: perform the counting procedure on the total effective filtration area;
- between 50 and 1 000 particles: perform the counting procedure on 20 randomly chosen unit areas and multiply the result by the appropriate factor;
- between 1 000 and 5 000 particles: perform the counting procedure on 10 randomly chosen unit areas and multiply the result by the appropriate factor;
- more than 5 000 particles: perform the counting procedure on 10 or fewer randomly chosen unit areas and multiply the result by the appropriate factor.

The number of unit areas chosen for the count shall be selected so that the number of the counted particles is not less than 300.

The measuring factor is the ratio between the effective filtration area and the total counted area (sum of the unit areas).

NOTE — When counting the particles at the boundary line of the selected area those at the upper and left edge zone are counted.

12.5.5 Expression and comparison of results

The results shall be expressed as the number of particles in each range per 100 ml of liquid. Record whether the microscopic examination was performed under incident or transmitted light.

Compare the table of results obtained with the applicable standard on the determination of the contamination level.

12.6 Test report

See section four, clause 15.

13 Method using an automatic particle-count instrument

13.1 Principle

Thorough mixing of the liquid sample to suspend all particles. Direct sizing and counting of solid particle contamination in the liquid sample using an automatic particle-count instrument.

The automatic particle counter used operates according to the principle of intermittent light-beam (light interruption technique). As soon as a specified liquid volume has passed the measuring area of the particle counter sensor, counting is terminated and results are recorded.

13.2 Apparatus

13.2.1 Automatic particle-count instrument, operating according to the principle of intermittent light-beam and equipped with an appropriate sensor for counting the required particle sizes according to the manufacturer's recommended procedure.

13.2.2 Compressed gas supply, air or nitrogen, supplying a consistent (but variable) gas flow at pressures up to 400 kPa (4 bar). The gas shall be free from water and oil and shall be filtered through a filter membrane having an aperture size less than or equal to 0,5 µm.

13.2.3 Sample bottles, as specified in clause 6.

The sample bottles shall not contain more than 200 particles greater than 10 µm nor more than 500 particles greater than 5 µm per 100 ± 5 ml.

13.2.4 Filter-jet spray gun (rinse dispenser), for the distribution of filtered solvent C (a pressure-operated system which discharges the solvent through a membrane filter, the aperture size of which is less than or equal to 0,5 µm).

13.3 Reagents and material

13.3.1 Solvent C (see clause 4), filtered.

13.3.2 Air Cleaner Fine Test Dust (ACFTD) contaminant, for instrument calibration in accordance with ISO 4402.

13.3.3 Latex spheres, for instrument calibration in accordance with ARP 1192 A.

The number and sizes as well as the tolerated deviation of these spheres shall be precisely defined by the producer and, if possible, certified by an independent institute.

13.4 Calibration of the particle-count instrument

IMPORTANT — The information obtained from automatic counters is directly related to the calibration and setting of the machine prior to use.

Care shall be taken to confirm the validity of the relative calibration before comparison is made with results obtained on other similar machines in other locations, otherwise the results cannot be related in absolute terms between different devices, such as microscopes counting the longest dimensions and automatic particle counters evaluating equivalent area or equivalent volume.

Calibration shall be carried out at least twice a year as well as at any time when the sensor or the electrical part of the instrument is altered. Calibration shall be carried out in accordance with one of the following methods:

a) using Air-Cleaner Fine Test Dust contaminant (13.3.2), method specified in ISO 4402;

b) using latex spheres (13.3.3), method specified in ARP 1192 A.

13.5 Procedure

13.5.1 Sample preparation

13.5.1.1 Counting shall, preferably, be carried out direct from the original sample bottle (13.2.3). If transfer cannot be avoided, the sample shall be thoroughly shaken. Perfectly clean environmental conditions are mandatory for this operation.

13.5.1.2 Samples showing contamination when visually inspected shall not be counted, as such contamination might adversely affect the proper performance of the sensor.

13.5.1.3 If the sample contains free water, it can be dried. If this does not succeed in eliminating the free water, counting shall not proceed.

13.5.1.4 If the counting capacity of the sensor is exceeded, it may become necessary to dilute the sample. However, this should be avoided whenever possible.

Either solvent C (13.3.1) or a liquid, preferably of the same type as or at least compatible with the sample, filtered through a membrane filter having an aperture size less than or equal to 1,2 µm, should be used for dilution.

13.5.1.5 Before counting, thoroughly shake the sample manually or mechanically, until the contaminants in the sample bottle are suspended homogeneously. Air bubbles can be removed by evacuation or ultrasonically.

13.5.2 Preparation of the particle-count instrument

13.5.2.1 The instrument shall be started up in accordance with the manufacturer's instructions. Ensure that the instrument has been switched on long enough to become stabilized.

13.5.2.2 Adjust the instrument to the calibration figures which have been determined during calibration of the instrument, in accordance with one of the methods specified in 13.4.

13.5.2.3 Set the particle-count instrument either for cumulative counting above a specific size (switch position: "Total") or for counting within specified size ranges (switch position: "Delta").

13.5.3 Counting procedure

13.5.3.1 Rinse the sensor with a small amount of the sample liquid.

13.5.3.2 Adjust the setting of the gas pressure in order to obtain the same flow volume as for the calibration.

13.5.3.3 Proceed with counting of the sample in accordance with the manufacturer's instructions.

13.5.3.4 Carry out at least two measurements with identical volumes per sample.

13.5.3.5 Once the counting procedure has been completed, remove the sample bottle and rinse the sensor with solvent C or a compatible fluid.

13.6 Expression of results

The results shall be expressed as the number of particles per 100 ml, either cumulatively above a specific size or within a specified size range.

The type of calibration means used for instrument calibration shall be included in the test report (see 15.3).

Example of tabulated results are given in tables 2a) and 2b).

Table 2a) — Example of tabulated results for counting within specified size ranges

Particle (p) size range µm	Number of particles per 100 ml
$5 < p \leqslant 15$	11 500
$15 < p \leqslant 25$	6 780
$25 < p \leqslant 50$	1 740
$50 < p \leqslant 100$	210
$100 < p$	15
Method used for calibrating instrument	ACFTD

Table 2b) — Example of tabulated results for cumulative counting (counting of total numbers)

Particle (p) size µm	Number of particles per 100 ml
$p > 5$	20 250
Method used for calibrating instrument	Latex spheres

13.7 Test errors

Data on repeatability and reproducibility cannot yet be given.

13.8 Test report

See section four, clause 15.

14 Gravimetric method

14.1 Principle

Filtering of a known volume of fluid under vacuum conditions through two identical superimposed membrane filters. The mass difference of both membranes after filtration represents the solid particle contamination content. The method is applicable to samples taken into sample bottles or directly into a field monitor.

14.2 Apparatus

14.2.1 Filtration apparatus, including

— a funnel, glass or stainless steel, approximately 250 ml capacity;

— a clamping device;

— a glass or stainless steel support, including a sintered glass or stainless steel filter-holder screen.

14.2.2 Funnel cover (e.g. an element of a Petri dish).

14.2.3 Membrane filters (pre-weighed or match-weight).

Diameter, aperture size and type of membrane filter shall be stated in the test report (see 15.3).

14.2.4 Vacuum flask.

14.2.5 Device for establishing a controllable vacuum.

14.2.6 Filter-jet spray gun (rinse dispenser), for the distribution of filtered solvent C (a pressure-operated system which discharges the solvent, in the form of a fine jet, through a membrane filter, the aperture size of which is less than or equal to the aperture size of the test membrane).

14.2.7 Calibrated measuring cylinder.

14.2.8 Forceps, stainless steel, with unserrated tips.

14.2.9 Petri dish, with cover.

14.2.10 Analytical balance, with an accuracy of ± 0,07 mg and containing a provision for the dissipation of static electricity.

14.2.11 Non-ventilated drying chamber, with an adjustable temperature range from 90 °C to 105 °C.

14.2.12 Adaptor, for attachment of monitor (14.2.13) to vacuum flask (14.2.4).

14.2.13 Monitor (see 9.2.1).

NOTE — Prior to use, the filtration apparatus (14.2.1), funnel cover (14.2.2), forceps (14.2.8) and Petri dish (14.2.9) shall be cleaned in accordance with the procedure specified in 7.2.

14.3 Procedure

14.3.1 Procedure A (using sample bottles)

14.3.1.1 Blank test filtration

14.3.1.1.1 Using forceps, remove two membrane filters (14.2.3) from the storage container.

14.3.1.1.2 Using the analytical balance (14.2.10), weigh the membrane filters and mark them at a point outside the effective filtration area (using, for example, a ball-point pen) with the letters "E"[1] and "T"[1], respectively.

When using match-weight membrane filters, weighing is not necessary.

14.3.1.1.3 Place both membranes on the support of the filtration apparatus, with membrane "T" placed under membrane "E", lower the funnel and secure the whole assembly with the clamping device.

14.3.1.1.4 Rinse the funnel with filtered solvent C from the filter-jet spray gun (14.2.6).

14.3.1.1.5 Apply the vacuum device (14.2.5) slowly up to 87 kPa (0,87 bar) max.

14.3.1.1.6 After evacuation of the solvent, remove clamping device and funnel, turn off the vacuum.

14.3.1.1.7 Place the membranes side by side in the cleaned Petri dish.

14.3.1.1.8 Place the Petri dish, with its cover half-open, in the drying chamber (14.2.11) at 90 °C to 105 °C and leave it there for 30 min.

14.3.1.1.9 Place the Petri dish, with its cover half-open, beside the balance for 30 min (tolerated humidity absorption).

14.3.1.1.10 Weigh the membrane filters "E" and "T" separately and record the masses as m_E and m_T, respectively.

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[1] The letter "E" stands for "essai" (test filter) and the letter "T" stands for "témoin" (control filter).

The mass difference ($m_E - m_T$) shall not exceed 0,1 mg.

NOTE — When not using match-weight membrane filters, the mass of the membrane filters, as determined in 14.3.1.1.2, should be taken into consideration.

14.3.1.2 Sample filtration

The usual sample volume is 100 ± 5 ml; this volume, however, can be modified depending on the level of contaminant contained in the sample to be analysed.

The sample volume used for determination shall be stated in the test report.

14.3.1.2.1 Using forceps, place both membrane filters on the support of the filtration apparatus, with membrane "T" in the lower position. Lower the funnel and secure the whole assembly with the clamping device.

14.3.1.2.2 Shake the flask containing the sample and remove cap. Pour the required volume in the clean measuring cylinder (14.2.7).

14.3.1.2.3 Pour the contents of the measuring cylinder into the funnel.

14.3.1.2.4 Pour approximately 50 ml of filtered solvent C into the measuring cylinder. Shake and pour into the funnel. Replace the funnel cover.

14.3.1.2.5 Apply the vacuum slowly up to 87 kPa (0,87 bar) max. and filter until the liquid level in the funnel is approximately 10 mm.

14.3.1.2.6 Remove the cover, rinse the funnel side walls with filtered solvent C using the filter-jet spray gun and replace the cover.

14.3.1.2.7 After evacuation of the solvent, remove cover, clamping device and funnel.

14.3.1.2.8 With the membrane filters under vacuum conditions, rinse the upper membrane surfaces with concentric jets of solvent C from the filtered solvent distributor.

The purpose of this operation is to collect sediments in the middle of the membrane and to perfect the control filter rinsing.

14.3.1.2.9 Turn off the vacuum.

14.3.1.2.10 Follow the same procedure as specified in 14.3.1.1.7 to 14.3.1.1.9.

14.3.1.2.11 Weigh the membrane filters "E" and "T" separately and record the masses as m_{EA} and m_{TA} respectively.

14.3.2 Procedure B (using the monitor) — Sample filtration

14.3.2.1 Fit the adaptor (14.2.12) onto the stopper of the vacuum flask (14.2.4).

14.3.2.2 Remove the protective caps from the monitor (14.2.13). Place the monitor with the filter surface onto the adaptor.

14.3.2.3 Apply the vacuum slowly up to 87 kPa (0,87 bar) max. and evacuate the filtered solvent C (approximately 100 ml).

14.3.2.4 Turn off the vacuum.

14.3.2.5 Open the monitor and, using forceps, place the membrane filters carefully side by side into a cleaned Petri dish.

14.3.2.6 Mark the membrane filters with the letters "E" and "T", respectively.

14.3.2.7 Place the Petri dish, with its cover half-open, in the drying chamber at 90 °C to 105 °C and leave it there for 30 min.

14.3.2.8 Then place the Petri dish, with its cover half-open, beside the balance for 30 min (tolerated absorption of humidity).

14.3.2.9 Weigh the membrane filters "E" and "T" separately and record the masses as m_{EB} and m_{TB}, respectively.

14.4 Expression of results

14.4.1 Procedure A

The total solid particle contamination, expressed in milligrams per 100 ml of fluid, of the sample is given by the formula

$$(m_{EA} - m_E) - (m_{TA} - m_T)$$

where

m_{EA} is the mass of membrane filter "E" after the sample filtration (see 14.3.1.2.11);

m_E is the mass of membrane filter "E" after the blank test filtration (see 14.3.1.1.10);

m_{TA} is the mass of membrane filter "T" after the sample filtration (see 14.3.1.2.11);

m_T is the mass of membrane filter "T" after the blank test filtration (see 14.3.1.1.10).

If a sample volume other than 100 ± 5 ml is used, it shall be stated in the test report.

If the difference ($m_{TA} - m_T$) exceeds 0,5 mg, the result shall be rejected, and a new sample shall be taken.