INTERNATIONAL STANDARD

ISO 15167

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Petroleum products — Determination of particulate content of middle distillate fuels — Laboratory filtration method

Produits pétroliers — Détermination de la teneur en particules des distillats moyens — Méthode de filtration en laboratoire

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Contents

Page

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

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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 3.

Draft International Standards prepared 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.

International Standard ISO 15167 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants.*

Petroleum products — Determination of particulate content of middle distillate fuels — Laboratory filtration method

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of the particulate content of middle distillate fuels with a closed flash point of 38 °C or higher, determined in accordance with ISO 2719, ISO 3679 or ISO 13736. It is not applicable to light distillate fuels (gasolines) or to aviation fuels.

A gravimetric limitation of the contamination of middle distillate fuels used in diesel engines and domestic applications should be used for the control of filter plugging and other operational problems, and this procedure is applicable up to 25 g/m^3.

The precision of this procedure is only valid if the results are obtained strictly in accordance with the provisions of this International Standard, particularly in respect of the material used for the filter (see the note in 6.9), sample size and filtration of the complete sample.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 2719:1988, *Petroleum products and lubricants — Determination of flash point — Pensky-Martens closed cup method.*

ISO 3170:1988, *Petroleum liquids — Manual sampling.*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling.*

ISO 3679:1983, *Paints, varnishes, petroleum and related products — Determination of flashpoint — Rapid equilibrium method.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 4259:1992, *Petroleum products — Determination and application of precision data in relation to methods of test.*

ISO 13736:1997, *Petroleum products and other liquids — Determination of flash point — Abel closed cup method.*

3 Term and definition

For the purposes of this International Standard, the following term and definition applies.

3.1
particulate content
quantity of material retained on a filter medium of nominal porosity 0,8 µm, expressed as grams per cubic metre (g/m^3) of the sample, after being subjected to the provisions of this International Standard

4 Principle

A specified volume of sample is filtered through a preweighed test membrane filter and the increase in membrane filter mass is determined after washing and drying. The change in mass of a control membrane filter located immediately below the test membrane filter is also determined. The particulate content is determined from the increase in mass of the test membrane filter relative to the increase in mass of the control membrane filter.

5 Reagents and materials

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade. Unless otherwise specified, use water of a purity equivalent to grade 3 of ISO 3696.

5.1 Propan-2-ol (isopropyl alcohol), commercial grade, filtered through a membrane filter of 0,45 µm or less nominal porosity.

5.2 Liquid detergent, water soluble.

5.3 Flushing fluid, heptane or 2,2,4-trimethylpentane, filtered through a membrane filter of 0,45 µm or less nominal porosity before use.

5.4 Tap water, assumed to be clean and generally potable. Where suitable supplies cannot be guaranteed, the available reticulated water shall be filtered as in 5.3, or commercially available non-carbonated bottled water used as a substitute.

6 Apparatus

6.1 Analytical balance, with a single- or double-pan and a weighing accuracy of 0,1 mg or better.

6.2 Air ionizer, to be used in the balance case.

NOTE 1 When using a solid pan balance, the air ionizer may be omitted provided that, when weighing a membrane filter, it is placed on the pan so that no part protrudes over the edge of the pan.

NOTE 2 Air ionizers should be replaced within one year of manufacture.

6.3 Oven, of the static type (without fan-assisted circulation), explosion-proof, capable of maintaining a temperature of 90 °C ± 5 °C.

6.4 Petri dishes, approximately 125 mm in diameter, with removable glass supports for membrane filters.

NOTE Small watch glasses have been found suitable for supporting the membrane filters.

6.5 Forceps, of stainless steel, flat-bladed, with non-serrated, non-pointed tips.

6.6 Laboratory tongs, of stainless steel or other suitable non-corrosive material, capable of handling container closures.

6.7 Vacuum system, of sufficient capacity to maintain an absolute pressure over the range of 1 kPa to 100 kPa.

6.8 Test membrane filters, plain, 47 mm in diameter, of nominal pore size 0,8 µm, composed of nylon or cellulose ester (see the note in 6.9).

6.9 Control membrane filters, 47 mm in diameter, of nominal pore size 0,8 µm, nylon or cellulose ester filters, either plain or gridded for identification.

NOTE The precision of this International Standard was obtained using only nylon filters.

6.10 Flushing fluid dispenser, normally fitted with a membrane filter of nominal pore size 0,45 µm or less, as shown in Figure 1.

NOTE A standard laboratory wash bottle may be used provided that the flushing fluid is pre-filtered through a membrane filter of nominal pore size 0,45 µm or less, and precautions are taken to maintain appropriate cleanliness of the interior of the wash bottle.

6.11 Filtration apparatus, as illustrated in Figure 2.

6.11.1 Filter funnel, consisting of a funnel and funnel base with a filter support such that a membrane filter can be gripped between the sealing surface and the base by means of a locking ring or spring-action clamp.

6.11.2 Receiving flasks, of 1 litre capacity, preferably graduated, to receive the filtered sample and flushing fluid washings separately (see 10.1 and 10.10). The flasks shall be of borosilicate glass, fitted with a sidearm to connect to the safety flask (6.11.3), and via this to the vacuum system. When used in the arrangement shown in Figure 2, they shall be protected against implosion.

Suitable protection measures against implosion include the use of a tight-fitting strong plastic mesh, or taping the flask to minimize the dispersion of glass splinters.

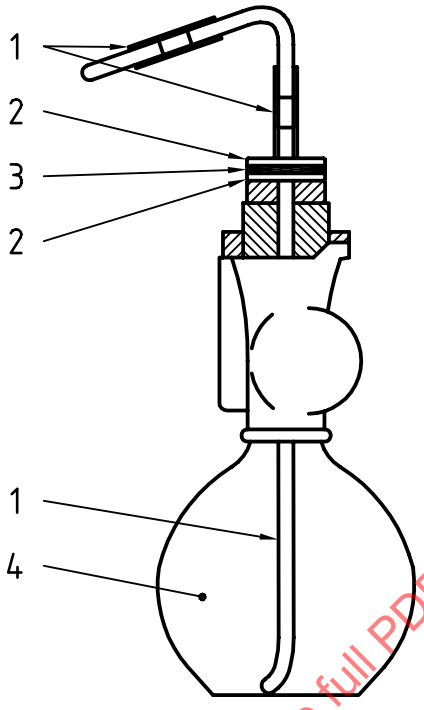
6.11.3 Safety flask, of minimum capacity 600 ml, of borosilicate glass, fitted with a sidearm connected to the receiving flask (6.11.2), and protected against implosion (see the preceding paragraph).

6.11.4 Ground/bond wire, of diameter 0,912 mm to 2,59 mm, bare, stranded, flexible, stainless steel or copper, installed in the flasks and grounded (earthed) as illustrated in Figure 2.

6.12 Plastic film, made of polyethylene or any other clear film that is resistant to the reagents and the sample.

NOTE Clean aluminium foil is also suitable.

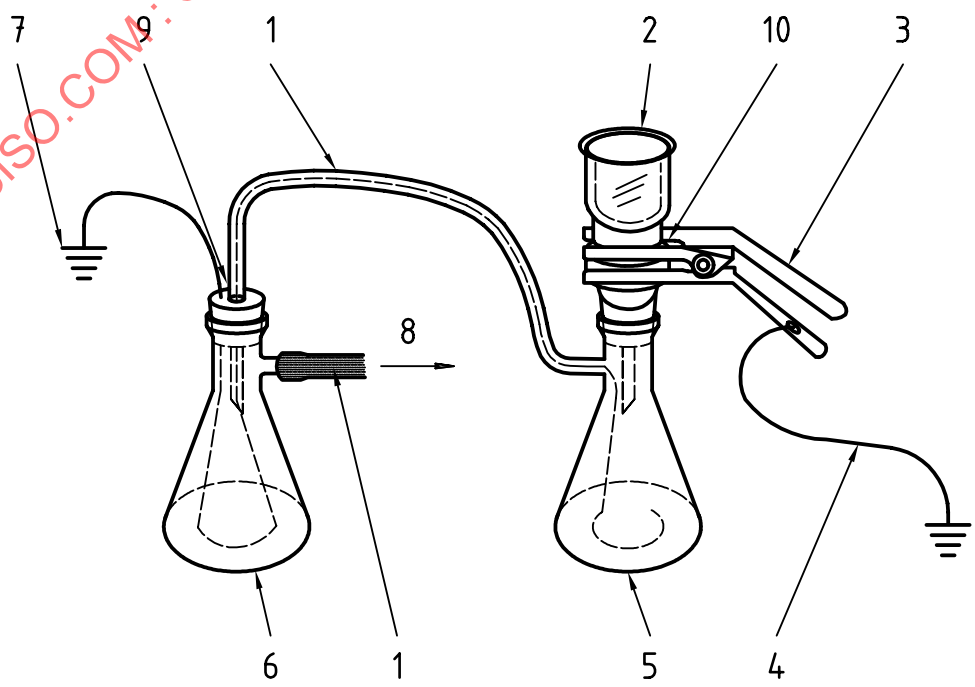
6.13 Graduated cylinders, made of borosilicate glass, of capacities between 100 ml and 500 ml.



Key

1 Reagent-resistant plastic tubing
2 Inert support screen
3 Membrane filter, 0,45 µm
4 Solvent-filtering dispenser

Figure 1 — Apparatus for filtering and dispensing flushing fluid



Key

1 Vacuum tubing
2 Funnel
3 Clamp
4 Wire to ground
5 Receiving flask
6 Safety flask
7 Laboratory ground (typical)
8 To vacuum pump
9 Insure seal between tube, hose and wire with appropriate sealant
10 File clamp jaws and handle where wire attaches to bare metal

Figure 2 — Apparatus for determining particulate content

7 Preparation of sample containers and apparatus

7.1 Clean strictly, in the manner described in 7.2 to 7.7, all the surfaces of all components of the sample containers (after removal of any labels, tags, etc.), sampling vessels, and parts of the apparatus that are

a) likely to come into contact with the sample or flushing fluid;

b) capable of transferring extraneous matter to the filter.

7.2 Wash with warm tap water (5.4) containing detergent (5.2).

7.3 Rinse thoroughly with warm tap water.

7.4 Rinse thoroughly with water, handling container caps only externally, with clean laboratory tongs (6.6) during this and subsequent washings.

7.5 Rinse thoroughly with propan-2-ol (5.1).

7.6 Rinse thoroughly with flushing fluid (5.3).

7.7 Cover the top of the sample container and the funnel opening of the assembled filtration apparatus with clean plastic film (6.12), previously rinsed with flushing fluid and air dried.

8 Samples and sampling

8.1 Ensure that only sampling equipment cleaned in accordance with clause 7 is used during sampling. As far as possible, sampling points and fixed equipment shall be brought to equivalent cleanliness. Every effort shall be made to avoid contamination from ambient conditions.

8.2 Use only containers of capacity 1 litre ± 0,15 litre.

8.3 Glass containers have an advantage in terms of the visibility of the interior for the efficiency of flushing, but clear glass containers risk particulate generation from ultraviolet light exposure. If clear glass containers are used, ensure that the sample receives the minimum exposure to light. Epoxy-lined cans, polytetrafluorethylene (PTFE) or high-density unpigmented linear polyethylene containers are also suitable.

8.4 The container shall be filled to 85 % to 95 % of its capacity.

8.5 Samples shall preferably be taken dynamically from a sampling loop in a distribution line or from the flushing line of a field sampling kit, in accordance with the principles specified in ISO 3171. Ensure that the line to be sampled is flushed with fuel before taking the sample.

8.6 Where it is only possible to obtain samples from static storage, follow the procedures given in ISO 3170, ensuring that the final sample has not passed through intermediate containers prior to placement in the prepared container.

NOTE Settlement of particulate matter is likely to result in erroneously low results on samples obtained from static storage. In order to avoid this, where possible, the contents of the tank should be circulated or agitated before sampling, or the sampling performed shortly after a tank has been filled.

9 Preparation of membrane filters

9.1 Prepare at least two pairs of membrane filters for each test, each pair consisting of a test membrane filter (6.8) and a control membrane filter (6.9).

9.2 Mark the Petri dishes (6.4) to identify the membrane filters.

9.3 Ensure that all glassware has been cleaned in accordance with clause 7.

9.4 Using forceps (6.5), place a test membrane filter (6.8) and a control membrane filter (6.9) side-by-side resting on glass supports in each Petri dish.

9.5 Place the Petri dishes, with their lids slightly ajar, in the oven (6.3) for 30 min.

9.6 Remove each Petri dish from the oven and place each dish near the balance (6.1). The dish covers shall remain ajar, but still protect the filters from contamination from the atmosphere. Allow 30 min for the membrane filters to come to equilibrium with room temperature and humidity.

9.7 Remove the control membrane filter from the Petri dish with the forceps (6.5), handling by the edge only, and place it centrally on the weighing pan of the balance. Weigh it, record the mass to the nearest 0,1 mg, and return it to the Petri dish.

9.8 Repeat 9.7 for the test membrane filter.

9.9 Using clean forceps (6.5), and handling by the edge only, place the weighed control membrane filter centrally in the filter support of the filtration apparatus (see Figure 2). Place the weighed test membrane filter on top of the control membrane filter. Install and clamp the funnel. Do not remove the plastic film from the funnel opening until ready to start filtration.

10 Procedure

10.1 It is likely that the entire contents of the sample container will be required to carry out a schedule of analyses following the determination of particulate content as specified in this International Standard. Therefore, it is essential that strict segregation is maintained between the glassware containing filtered sample and any that may contain traces of other materials such as flushing fluid. A fresh clean sample container, appropriately marked, shall be reserved for the rapid transfer of segregated filtered sample, in order that the risk of contamination leading to change in other required properties is minimized.

10.2 Thoroughly clean the outside of the sample container in the region of the cap by washing with detergent (5.2) in warm tap water (5.4), and rinsing with tap water followed by propan-2-ol (5.1). Discard the washings.

10.3 Shake the container vigorously for 30 s ± 5 s.

10.4 Remove the cap and get rid of any external contaminant that may be present in the internal threads and inside seal of the cap by washing with flushing fluid (5.3), ensuring that none of the washings enter the container. Place the washings in a clean dry container covered with plastic film (6.12).

10.5 Complete the assembly of the filtration apparatus (6.11) and place it in a fume cupboard.

10.6 Transfer fuel from the sample container to a 500 ml graduated cylinder (6.13), record the volume, start the vacuum and then transfer fuel in stages to the filter funnel.

NOTE Most fuels will filter reasonably rapidly during transference of the total content of the sample container. However, some fuels, due to the quantity and/or nature of particulates, may plug the membrane during filtration. If filtration slows, it is advisable to use smaller graduated cylinders of 100 ml capacity.

10.7 If the fuel is still flowing rapidly after the first transfer, repeat 10.6 until all of the liquid in the sample container has been transferred, and the volume measured.

10.8 Disconnect the receiving flask, and transfer the filtered sample rapidly into a clean dry sample container (see 10.1).

10.9 If filtration slows so that 100 ml of sample requires greater than 10 min for complete filtration, record the volume filtered, follow 10.8 and then 10.10. Repeat 10.6 and 10.7 using a second pair of membrane filters inserted into the filtration apparatus as specified in 9.9. Repeat again as necessary.

NOTE As soon as it is noted that filtration is slowing (see the note in 10.6), it is recommended that transfer to the filter funnel is reduced to small increments, in order to minimize liquid above the filter media at a time of blockage.

10.10 If the test portion in the filter funnel stops flowing completely whilst there is still liquid above the filter media, proceed as described in 10.10.1. If the flow of liquid has slowed below that required in 10.9, but the liquid above the filter media has all been filtered, proceed as described in 10.10.2.

10.10.1 Disconnect the receiving flask from the vacuum and remove the filter funnel assembly carefully, maintaining the filter funnel upright. Replace the first filter funnel assembly on the flask by a second (or more) filter funnel assembly complete with a fresh pair of weighed membrane filters. Carefully decant the liquid above the filter media in the first assembly into the second filter funnel assembly. Reconnect the vacuum to the original flask with the new filter funnel assembly connected, and continue the filtration. Connect the first filter funnel assembly to another receiving flask and proceed as described in 10.10.2.

10.10.2 Replace the receiving flask with another (not necessarily to the same standard of cleanliness), and wash down the inside of the funnel and the outside of the joint between the funnel and the filter base with flushing fluid (5.3). With the vacuum applied, carefully separate the funnel from the filter base and wash the periphery of the membrane filter by directing a gentle stream of flushing fluid from the edge to the centre, taking care not to wash any of the particulate from the surface of the membrane filter. Maintain the vacuum after the final washing for 10 s to 15 s, or until all excess flushing fluid is removed from the membrane filter.

Using clean forceps (6.5), carefully remove the test and control membrane filters from the filter base and place them in a clean Petri dish. Dry and reweigh the filters as described in 9.5 to 9.7, taking great care not to disturb the particulate on the surface of the test membrane filter.

10.11 When all the liquid in the sample container has been filtered, record the total volume filtered and replace the receiving flask with another (not necessarily to the same standard of cleanliness), and thoroughly rinse the sample container and graduated cylinder(s) with flushing fluid (5.3). Use three rinses of 25 ml each for each container, and pour the rinses through the filter funnel. Ensure that the containers are totally cleansed of particulate. Also pour through the filter funnel the washings obtained in 10.4, rinsing the container with one portion of 25 ml of flushing fluid. Carry out the washing, disassembly, drying and weighing procedure specified in 10.10.2.

11 Calculation

Calculate the particulate content, P, in grams per cubic metre, using the following equation:

$$P = \frac{(m_2 - m_1) - (m_4 - m_3)}{(V)} \times 10^6$$

where

m_1 is the initial mass of the test membrane filter, in grams;

m_2 is the final mass of the test membrane filter, in grams;

m_3 is the initial mass of the control membrane filter, in grams;

m_4 is the final mass of the control membrane filter, in grams;

V is the total volume of the sample filtered, in millilitres.

12 Expression of results

Report the result as the particulate content, in grams per cubic metre, to the nearest 0,1 g/m^3, together with the total volume, in millilitres, of sample filtered during the test. If more than one pair of membrane filters was used, the number of pairs shall be included in parentheses.

NOTE A typical reporting format would be:

Particulate content, g/m^3/ml 14,2/985 (3)