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Petroleum products — Determination of the filterability of lubricating oils —

Part 1: Procedure for oils in the presence of water

Produits pétroliers — Détermination de la filtrabilité des huiles lubrifiantes —

Partie 1: Méthode pour les huiles en présence d'eau

Reference number
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Contents

Page

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 28, *Petroleum and related products: fuels and lubricants from natural or synthetic sources.*

This second edition cancels and replaces the first edition (ISO 13357-1:2002), of which it constitutes a minor revision including alternative membranes in order to enable the continued use of this document.

A list of all parts in the ISO 13357 series can be found on the ISO website.

Introduction

As the fluid in a hydraulic system acts as a lubricant to minimize wear of the components, it is important to reduce the concentrations of circulating hard contaminant particles. This is particularly necessary when the performance of the system depends on the maintenance of small clearances and orifices. Removal of these contaminants is effected by the use of filters. The ability of a hydraulic fluid to pass through fine filters, without plugging them, is called its "filterability". This document describes a laboratory test procedure for assessing the filterability of mineral oils which have been heat-soaked in the presence of water. Filterability so determined is not a physical characteristic of the oil, but represents an estimation of its behaviour in service.

This document describes two measurements, referred to as "stages". The Stage I determination is based on a comparison of the mean flow rate of a fluid through a test membrane with its initial flow rate. Oils having good Stage I filterability, but only a poor Stage II performance (see below), would be unlikely to give performance problems in use, unless extremely fine system filters are utilized.

The Stage II determination is based upon the ratio between the initial flow rate of fluid through the test membrane and the rate at the end of the test. It is considered that this part of the procedure is a more severe test, and is more sensitive to the presence of gels and fine silts in the oil. Silts and gels may be present in an oil when it is produced, or could be formed as an oil ages, especially when hot. An oil with good Stage II filterability would be unlikely to give filtration problems even in the most extreme conditions, and with fine (less than 5 µm) filtration present. It would thus be suitable for use in more critical hydraulic and lubrication systems.

The procedure has been evaluated with mineral oils up to ISO viscosity grade 100. There would appear to be no reason why it should not be used with oils of higher viscosity grade (ISO viscosity grade 220 is a practical maximum), but the data obtained could not be claimed to be completely in accordance with this method. Similarly, it should be possible to extend the test procedure to fluids other than mineral oils. However, some fluids, e.g. fire-resistant fluids, will not be compatible with the specified test membranes, and the test could only be used for comparison purposes even when suitable membranes, with similar pore size/pore density characteristics to those specified in this procedure, have been identified.

Petroleum products — Determination of the filterability of lubricating oils —

Part 1:
Procedure for oils in the presence of water

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

1 Scope

This document specifies a procedure for the evaluation of the filterability of lubricating oils, particularly those designed for hydraulic applications, in the presence of water. The procedure only applies to mineral-based oils, since fluids manufactured from other materials (e.g. fire-resistant fluids) may not be compatible with the specified test membranes. The range of application has been evaluated with oils of viscosity up to ISO viscosity grade (VG) 100, as defined in ISO 3448. Within the range described, the filterability as defined is not dependent on the viscosity of the oil. The procedure is not suitable for some hydraulic oils on which specific properties have been conferred by the use of insoluble/partially soluble additives, or by particularly large molecular species.

NOTE Filterability is a prime requirement for lubricating oils used in hydraulic systems because of the fine filters used in this application.

This document defines a method for assessing the filterability of oils in the presence of contaminating water. It is noted that some oils will exhibit poorer filterability characteristics in these conditions. ISO 13357-2[1] is used to investigate the filterability of an oil which is used in applications where the presence of water in the oil is unlikely. An oil which has good filterability in the presence of contaminating water will not necessarily have equally good filterability in dry conditions. An oil having good filterability only when wet is unlikely to be generally acceptable.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1219-1, *Fluid power systems and components — Graphical symbols and circuit diagrams — Part 1: Graphical symbols for conventional use and data-processing applications*

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3448, *Industrial liquid lubricants — ISO viscosity classification*

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

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

ISO 6614:1994, *Petroleum products — Determination of water separability of petroleum oils and synthetic fluids*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
filterability
dimensionless number which is the ratio, expressed as a percentage, between volumes (Stage I) or flow rates (Stage II) at specified intervals in the test procedure

3.2
stage I filterability
ratio, expressed as a percentage, between 240 ml and the volume of oil actually filtered in the time that 240 ml would have theoretically taken, assuming no plugging of the membrane

3.3
stage II filterability
ratio, expressed as a percentage, between the flow rate near the start of the filtration, and the flow rate between 200 ml and 300 ml of filtered volume

4 Principle

The test fluid is treated with water at an elevated temperature, filtered under specified conditions through a membrane of 0,8 µm mean pore diameter, and the times for the specified filtrate volumes are recorded. Filterabilities are calculated from ratios of the filtration rate near the start of filtration to the filtration rate at specified higher filtered volumes. The result of the test is the average of three determined values.

NOTE In the ideal situation, the filtration rate remains constant.

5 Reagents and materials

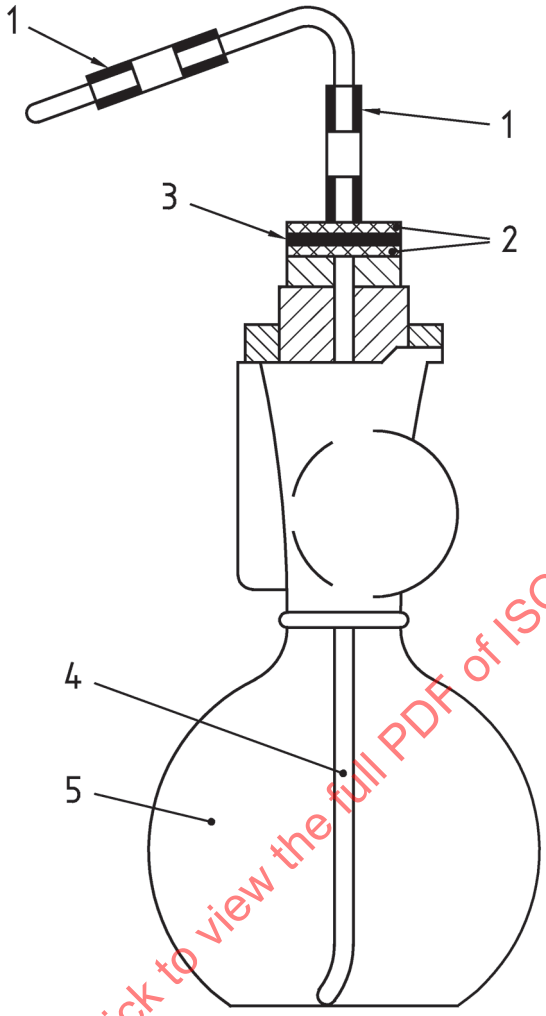
5.1 Water, conforming to grade 3 of ISO 3696.

5.2 Propan-2-ol (isopropyl alcohol), filtered through a compatible 0,45 µm membrane filter.

NOTE A solvent-filtering dispenser, as shown in Figure 1, is a means of dispensing this solvent, and the wash solvent (5.3).

5.3 Wash solvent, of light aliphatic hydrocarbon, filtered through a compatible 0,45 µm membrane filter (see NOTE under 5.2). Heptane or 2,2,4-trimethylpentane is suitable.

5.4 Compressed gas, complete with regulator system capable of supplying gas at nominated pressures between 50 kPa and 200 kPa. The gas (air or nitrogen) shall be dry and filtered.



Key

1	reagent-resistant plastic tubing	4	reagent-resistant plastic tubing
2	inert support screen	5	solvent-filtering dispenser
3	membrane filter, 0,45 µm		

Figure 1 — Solvent-filtering dispenser

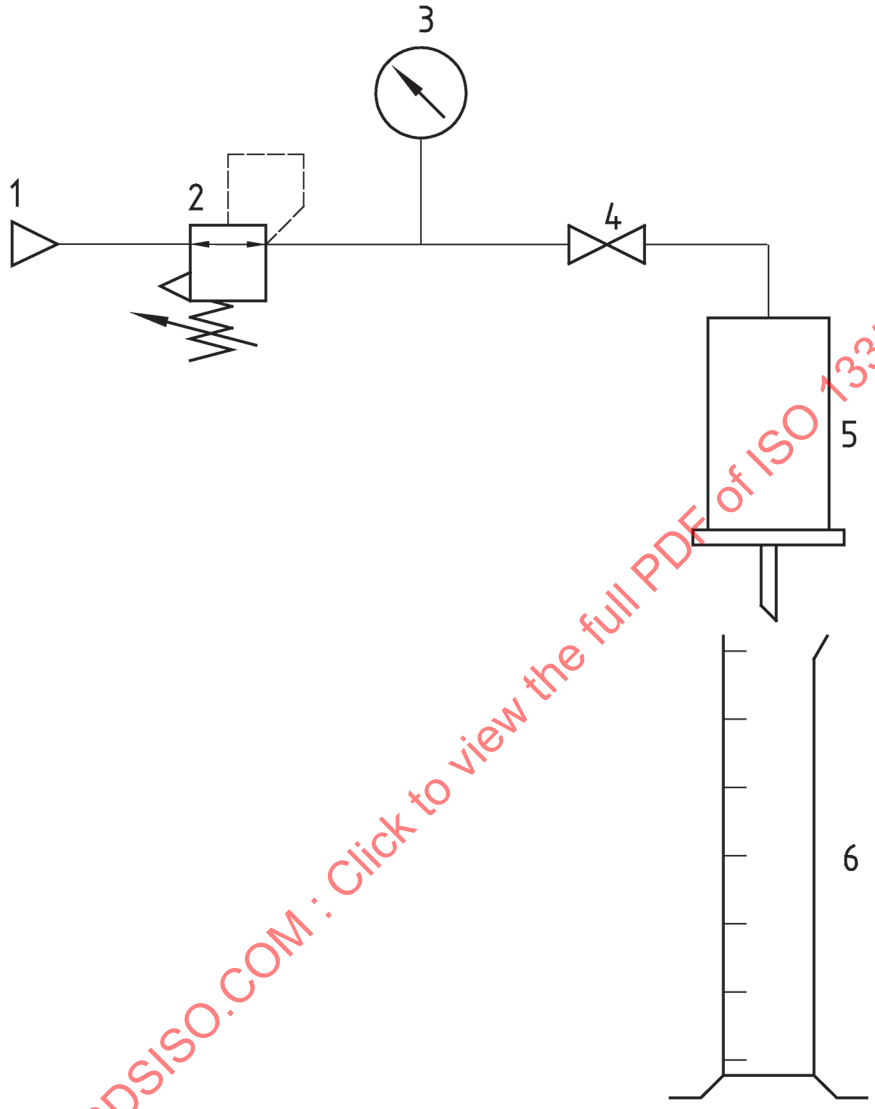
6 Apparatus

6.1 Filtration apparatus, constructed of stainless steel, consisting of a lidded funnel of at least 350 ml capacity, and a funnel base with filter support, such that a membrane filter (6.2) can be clamped between the sealing surfaces of the funnel and the base by means of a metal clamp or other suitable gas-tight closure. The apparatus shall be grounded (earthed), and suitable electrical bonding of the parts shall be provided. The effective filtration area shall be 1 130 mm^2 ± 60 mm^2. A schematic of the assembled apparatus, with the graphic symbols conforming to ISO 1219-1, is shown in Figure 2.

6.2 Membrane filters, of mixed cellulose esters, diameter 47 mm and mean pore size of 0,8 µm.

NOTE Millipore membranes of an equivalent specification to their filter membranes, catalogue number AAWP04700[1)], have been found satisfactory.

All the membranes used for a single test (3 determinations) should be taken from a single box. If membranes are taken from more than one box, all boxes shall be from the same production batch.



Key

1	source of compressed air or nitrogen	4	ball valve
2	pressure regulator	5	pressure vessel with membrane support
3	pressure gauge	6	measuring cylinder

Figure 2 — Outline of assembled filtration apparatus

6.3 Measuring cylinders, one of borosilicate glass, of 250 ml capacity, conforming to the requirements of ISO 4788. This cylinder shall be permanently marked with further graduation marks at 10 ml and 300 ml. Annex A describes a procedure for adding these graduations. A second cylinder, capable of measuring 330 ml ± 5 ml, is also required for sample transfer.

1) Millipore membrane, catalogue number AAWP04700, is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

NOTE 1 The 250 ml measuring cylinder has a total capacity in excess of 300 ml, allowing the extra graduations to be added. The use of a larger measuring cylinder for the filtration process would not give adequate precision for the test.

NOTE 2 It is advantageous, particularly with oils having very low electrical conductivity (e.g. ashless oils), to wrap the cylinder with a grounded (earthed) metal helix or mesh which does not obscure the graduations.

6.4 Pressure gauge, dial or digital type, capable of reading the required delivery pressures (see 9.12) ±5 kPa.

6.5 Forceps, spade-ended.

6.6 Timing device, electronic or mechanical, capable of reading to the nearest 0,2 s, and fitted with a dual-stop facility.

6.7 Oven, controlled at 70 °C ± 2 °C.

6.8 Petri dishes, loosely covered.

6.9 Bottles, of 500 ml laboratory type with screw caps. The exact shape of the bottle is unimportant, and 500 ml conical flasks can be used. However, the neck should be fairly narrow, but shall be wide enough to accept the stirrer (6.10). It is essential that the base of the bottle be fairly flat.

6.10 Motor and stirrer, conforming to the requirements of ISO 6614:1994, 6.3.

6.11 Pipettes.

6.11.1 Pasteur or dropping pipette.

6.11.2 1 ml graduated pipette.

7 Samples and sampling

7.1 Unless otherwise specified, samples shall be taken by the procedure specified in ISO 3170.

7.2 Shake the laboratory sample thoroughly by hand, and allow it to stand for 24 h at a temperature of 15 °C to 25 °C. The laboratory temperature should not vary by more than ±2 °C for the duration of the test.

NOTE The optimum ambient laboratory temperature for precision is 22 °C.

8 Preparation of apparatus

8.1 Rinse the apparatus with wash solvent (5.3) to remove traces of oil from previous tests.

8.2 Soak in laboratory detergent solution overnight, or scrub thoroughly with hot laboratory detergent solution.

8.3 Rinse with hot tap water, followed by cold tap water.

8.4 Rinse with water (5.1).

8.5 Rinse with propan-2-ol (5.2).

8.6 Rinse with wash solvent (5.3) and allow to dry.

9 Procedure

9.1 A diagram of a typical determination is shown as Figure 3.

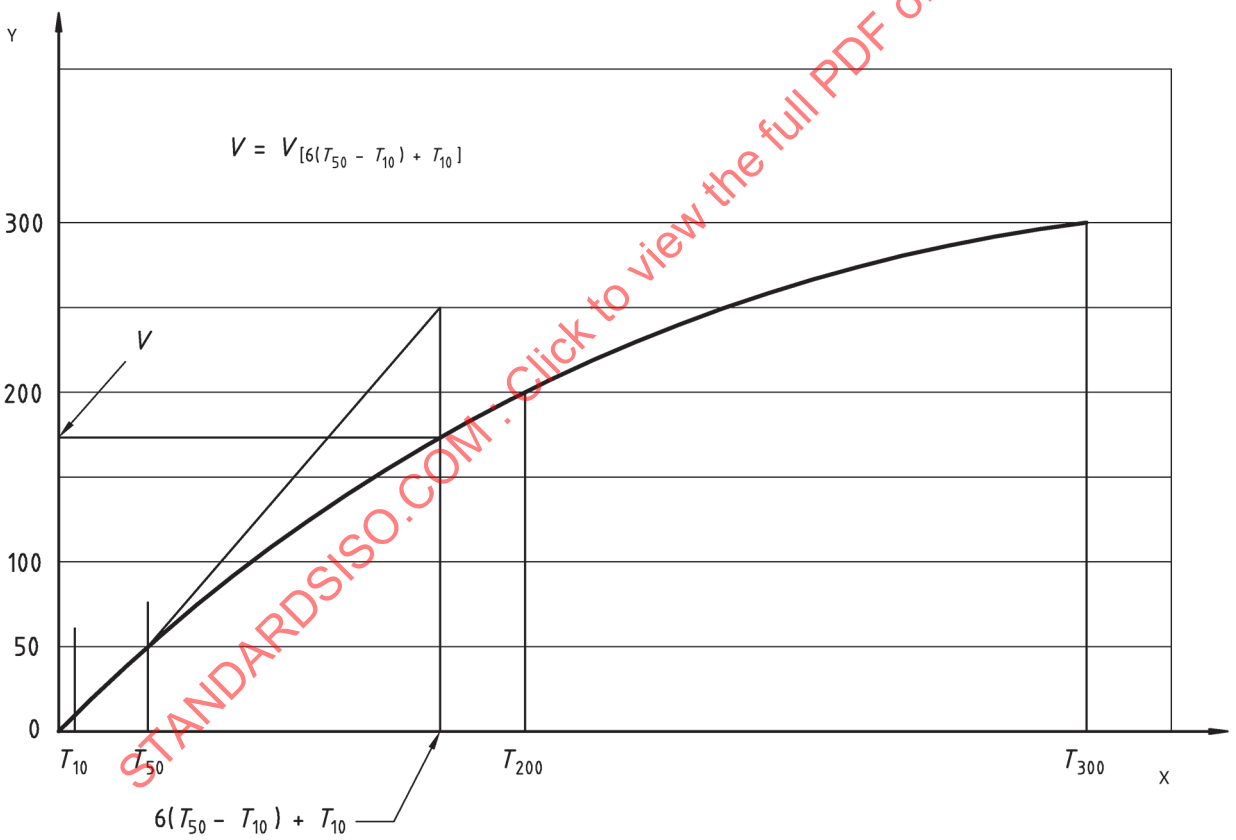
9.2 Carry out the test in triplicate. Between each of the three tests rinse the apparatus with wash solvent (5.3) and allow to dry.

9.3 Mix the laboratory sample by inverting the sample container sharply 30 times in 60 s ± 10 s. Each inversion should be completed by a distinct snap.

9.4 Measure 330 ml ± 5 ml of sample by means of the measuring cylinder (6.3), and transfer to a bottle (6.9).

9.5 Using a graduated pipette (6.11.2), add 0,66 ml ± 0,02 ml of water (5.1).

A suitable syringe or micropipettor may be used to add the water, provided it has sufficient accuracy.



Key

X time, s

Y volume, ml

Figure 3 — Diagram of typical filterability test run

9.6 Stand the bottle, with the cap slightly loosened to avoid a build-up of pressure, in the oven (6.7) for 2 h ± 5 min.

9.7 Remove the bottle from the oven and stir the contents using the stirrer (6.10) for 5 min ± 2 s. The stirrer paddle shall be maintained 5 mm ± 1 mm above the bottom of the bottle. Check immediately after starting the stirrer that the shaft speed is 1 500 r/min ± 50 r/min.

9.8 Return the bottle, with the cap slightly loosened, to the oven for 70 h ± 1 h. Tighten the cap after approximately 30 min to avoid water loss.

9.9 Remove the bottle from the oven and keep it at laboratory ambient temperature, in the dark, for a further 24 h ± 2 h. Ideally, the preparation of all three water-treated samples should be carried out during one day, so that the filtration steps given in 9.10 to 9.23 can be performed four days later.

9.10 Place a membrane filter (6.2) in a loosely covered Petri dish (6.8) in the oven (6.7) for 10 min. Handle the membrane filter by the edge only, using forceps (6.5), during this and all subsequent operations.

9.11 Assemble the filtration apparatus (6.1) with a membrane filter the correct way up in place. The correct orientation of the membrane filters is that in which the top is viewed on the normal opening of the packaging box.

Ensure that the apparatus is properly grounded (earthed), there are no leaks in the pressure system, and that the measuring cylinder (6.3) is properly located below the filtration vessel.

With oils having very low electrical conductivity (e.g. ashless oils), it may be advantageous for the tip of the outlet of the pressure vessel (6.1) to be just below the lip of the measuring cylinder (6.3).

9.12 Close the ball valve (see Figure 2) and adjust the gas pressure to the specified level, according to the viscosity of the oil (determined in accordance with ISO 3448). The required pressures, ±5 kPa, are

— ISO viscosity grades (VG) less than 32 50 kPa,

— ISO viscosity grades (VG) of 32 and 46 100 kPa,

— ISO viscosity grades (VG) of 68 and 100 200 kPa.

9.13 Using the pipette (6.11.1), add only enough laboratory sample (7.2) to completely wet the membrane with oil.

9.14 Mix the sample (9.9) by inverting the bottle vigorously 30 times in 60 s ± 10 s. Each inversion should be completed by a distinct snap.

9.15 Immediately pour the entire contents of the bottle into the filtration funnel and close and seal the lid. Open the ball valve and check immediately that the correct pressure is maintained.

9.16 Start the timing device (6.6) when the first drop of oil enters the measuring cylinder.

9.17 Using the dual-stop facility of the timing device, record, to the nearest 0,2 s, the time when the level in the measuring cylinder reaches 10 ml (T_{10}), 50 ml (T_{50}), 200 ml (T_{200}) and 300 ml (T_{300}).

9.18 When T_{50} is available, calculate T_V as 6 ($T_{50} - T_{10}$) + T_{10}.

9.19 Record the volume in the measuring cylinder at the time (T_V) calculated in 9.18.

9.20 If Stage I filterability only is being measured, discontinue the test when this volume has been recorded.

9.21 Discontinue the test if the time to the highest required volume (either T_V or T_{300}) exceeds 7 200 s (2 h).

9.22 After the time to the highest required volume has been recorded, close the ball valve and the gas supply valve, depressurize the apparatus and remove the funnel.

9.23 Visually inspect the membrane filter for homogeneity of coloration. Repeat the determination if the colour of the membrane filter is visually significantly uneven.

NOTE An uneven colour indicates that not all of the membrane surface was used in the filtration of the oil, and the results are unlikely to be reproducible.

10 Calculations

10.1 Stage I filterability

Calculate the Stage I filterability F_I from Formula (1):

$$F_I = \frac{V - 10}{240} \times 100 \quad (1)$$

where

V is the volume collected at T_V (see 9.19), in millilitres;

T_V is 6 (T_{50} – T_{10}) + T_{10} (see 9.18), in seconds.

10.2 Stage II filterability

Calculate the Stage II filterability F_{II} from Formula (2):

$$F_{II} = \frac{2,5\left(T_{50} - T_{10}\right)}{T_{300} - T_{200}} \times 100 \quad (2)$$

11 Expression of results

11.1 General

The results of the test (Stage I and/or Stage II) will normally be reported as “pass” or “fail” against a limiting value of 50. Pass results above 50 may be accompanied by the average determined value in parentheses. Results below 50 generally have poor reproducibility, and the average value should not be reported.

11.2 Assessment of validity

11.2.1 If the three determined values are above 50, report the result as “pass” and optionally report the average of the three determinations for the assessment of filterability, to the nearest whole number. If all three results are below 50, report the result as “fail”.

11.2.2 If two of the determined values are above 50, and the third is below, but not by more than 10, then report the result as “pass”. Also, report the fact that only two of the values were above 50.

11.2.3 If two of the determined values are below 50, but one is above 60, repeat the test.

11.2.4 Report the result as "unfilterable" if the filtration time exceeds 7 200 s (2 h).

12 Precision

Precision is not obtainable on tests with a simple pass/fail assessment, "<50" indicating a "fail" in this context. Inter-laboratory results on the procedure described in this document show that, at values below approximately 50, the scatter of results is poor.

Where the filterability is higher, the scatter of results improves in line with rising values of filterability, and the estimated precision values given in ISO 13357-2 may be used as a guide.

13 Test report

The test report shall contain at least the following information:

a) a reference to this document, i.e. ISO 13357-1;

b) the type and complete identification of the product tested;

c) the results of the test (see Clause 11);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of test.

Annex A
(informative)

Suitable procedure for the addition of graduations to a measuring cylinder

A.1 Apparatus

A.1.1 **Graduated cylinder**, of 250 ml nominal capacity, as described in 6.3.

A.1.2 **Syringe**, fitted with a sufficient length of fine-bore tubing to reach nearly to the bottom of the cylinder (6.3) with a capacity of approximately 20 ml.

A.1.3 **Oven** or **furnace**, controlled to 300 °C or 500 °C as appropriate.

A.1.4 **Balance**, capable of weighing up to 500 g with an accuracy of ±0,1 g.

A.1.5 Either **paint pen,** fine-tipped[2)], for marking glassware.

A.1.6 Or **decals**, ceramic, for firing onto glassware.

A.2 Procedure

A.2.1 Ensure that the graduated cylinder is completely clean and dry.

A.2.2 Fill the cylinder (A.1.1) carefully to the 50 ml mark with water (5.1), ensuring that the bottom of the meniscus is level with the middle of the 50 ml graduation. Avoid wetting the cylinder above the final water level during this operation.

A.2.3 Weigh the cylinder, to the nearest 0,1 g, and remove water from the cylinder via the syringe and tubing (A.1.2) until 40 g ± 0,1 g has been removed. Allow the water to drain down to its new level.

A.2.4 Follow either A.2.4.1 or A.2.4.2.

A.2.4.1 Stand the cylinder on the workbench and clamp the pen (A.1.5) horizontally so that the centre of the tip is in line with the bottom of the meniscus. Rotate the cylinder against the tip to mark the glass with a painted line.

A.2.4.2 Apply the ceramic decal (A.1.6) so that the middle of the mark is in line with the bottom of the meniscus.

A.2.5 Fill the cylinder carefully to the 250 ml mark with water, as in A.2.2.

A.2.6 Weigh the cylinder to the nearest 0,1 g and add water to the cylinder until 50,0 g ± 0,1 g has been added. Avoid wetting the cylinder above the final water level during this operation.

2) A “Sharpie” ‘Oil-Based Paint Marker - Extra Fine Point’ is suitable for this purpose. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.