

Petroleum products — Determination of the filterability of lubricating oils —

Part 1: Procedure for oils in the presence of water

ICS 75.100

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Summary of pages

This document comprises a front cover, an inside front cover, the ISO title page, pages ii to iv, pages 1 to 9 and a back cover.

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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



Foreword

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International Standard ISO 13357-1 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

ISO 13357 consists of the following parts, under the general title *Petroleum products — Determination of the filterability of lubricating oils*:

- *Part 1: Procedure for oils in the presence of water*
- *Part 2: Procedure for dry oils*

Annex A of this part of ISO 13357 is for information only.

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 part of ISO 13357 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 part of ISO 13357 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 on 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 or 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 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 part of ISO 13357 may involve hazardous materials, operations and equipment. This part of ISO 13357 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 13357 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 13357 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 part of ISO 13357 defines a method for assessing the filterability of oils in the presence of contaminating water. It should be noted that some oils will exhibit poorer filterability characteristics in these conditions. ISO 13357-2 should be used to investigate the filterability of an oil which is to be 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 normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 13357. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 13357 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 1219-1:1991, *Fluid power systems and components — Graphic symbols and circuit diagrams — Part 1: Graphic symbols*

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

ISO 3448:1992, *Industrial liquid lubricants — ISO viscosity classification*

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

ISO 13357-1:2002(E)

ISO 4788:1980, *Laboratory glassware — Graduated measuring cylinders*

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

ISO 13357-2:1998, *Petroleum products — Determination of the filterability of lubricating oils — Part 2: Procedure for dry oils*

3 Terms and definitions

For the purposes of this part of ISO 13357, the following terms and definitions apply.

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.

Ideally, the filtration rate should remain constant.

5 Reagents and materials

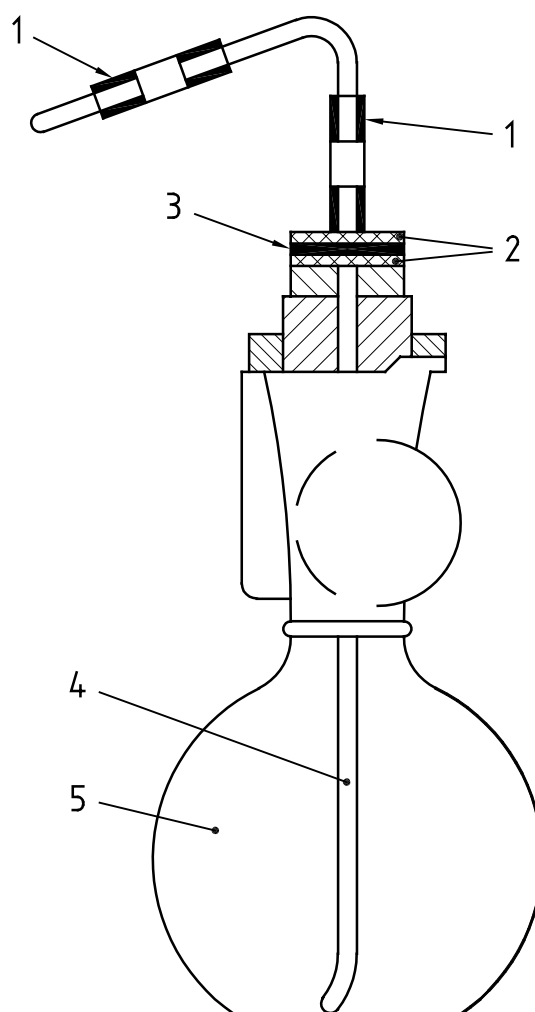
5.1 Water, conforming to grade 3 of ISO 3696.

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 the note to 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 of between 50 kPa and 200 kPa. The gas (air or nitrogen) shall be dry and filtered.



Key

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

Figure 1 — Solvent-filtering dispenser

6 Apparatus

A schematic of the assembled apparatus, with graphic symbols in accordance with ISO 1219-1, is shown in Figure 2.

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 $1\,130\text{ mm}^2 \pm 60\text{ mm}^2$.

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

NOTE Membranes of an equivalent specification to Millipore filter membranes, catalogue number AAWP 047 IF, have been found satisfactory.

6.3 Measuring cylinders, one of borosilicate glass, of 250 ml capacity, conforming to the requirements of ISO 4788 (see A.1.1). 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 \pm 5 ml, is also required for sample transfer.

NOTE 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.

6.4 Pressure gauge, dial or digital type, capable of reading the required delivery pressures (see 9.12) \pm 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 \pm 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 may 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, subclause 6.3.

6.11 Pipettes

6.11.1 Pasteur or dropping pipettes.

6.11.2 1 ml graduated pipettes.

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 \pm 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.

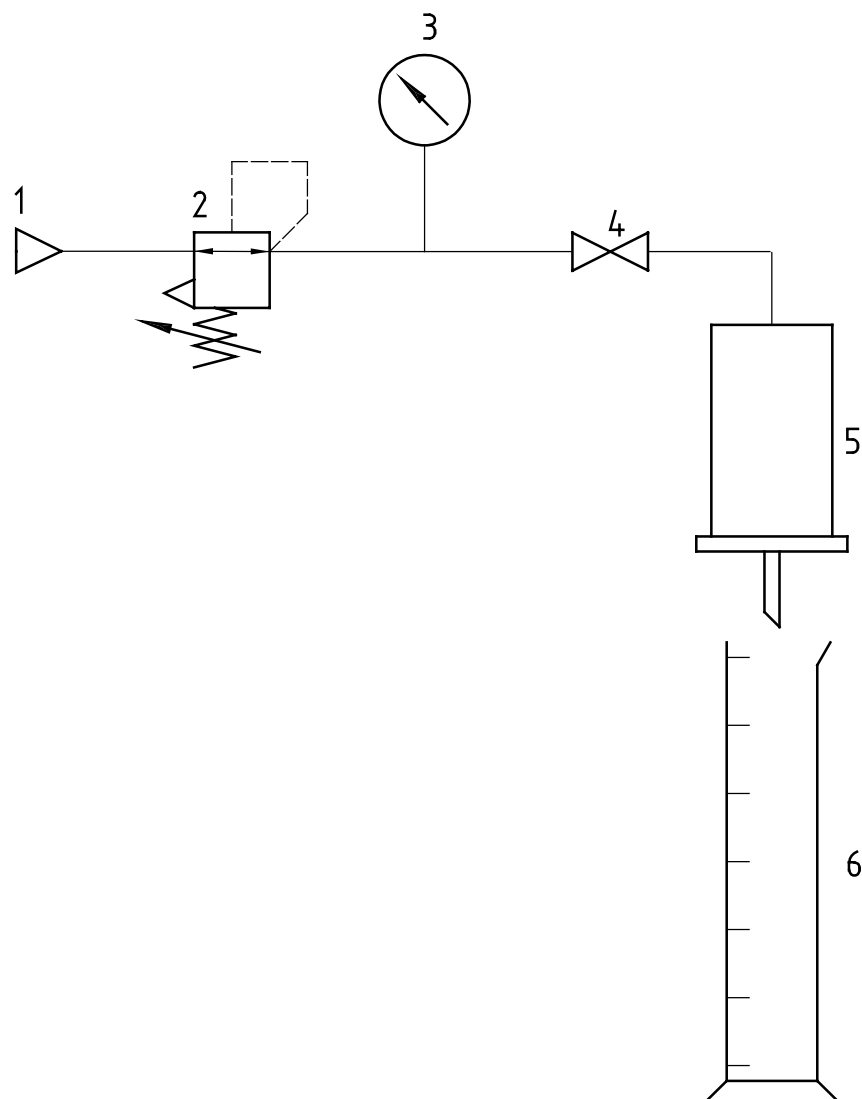
8.2 Soak in laboratory detergent solution overnight, or scrub thoroughly with hot laboratory detergent solution.

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.

**Key**

- | | |
|---|---------------------------------------|
| 1 | Source of compressed air or nitrogen |
| 2 | Pressure regulator |
| 3 | Pressure gauge |
| 4 | Ball valve |
| 5 | Pressure vessel with membrane support |
| 6 | Measuring cylinder |

Figure 2 — Outline of assembled filtration apparatus**9 Procedure**

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

9.2 Carry out the test in triplicate.

9.3 Mix the laboratory sample by inverting the sample container sharply 30 times in $60 \text{ s} \pm 10 \text{ s}$. Each inversion should be completed by a distinct snap.

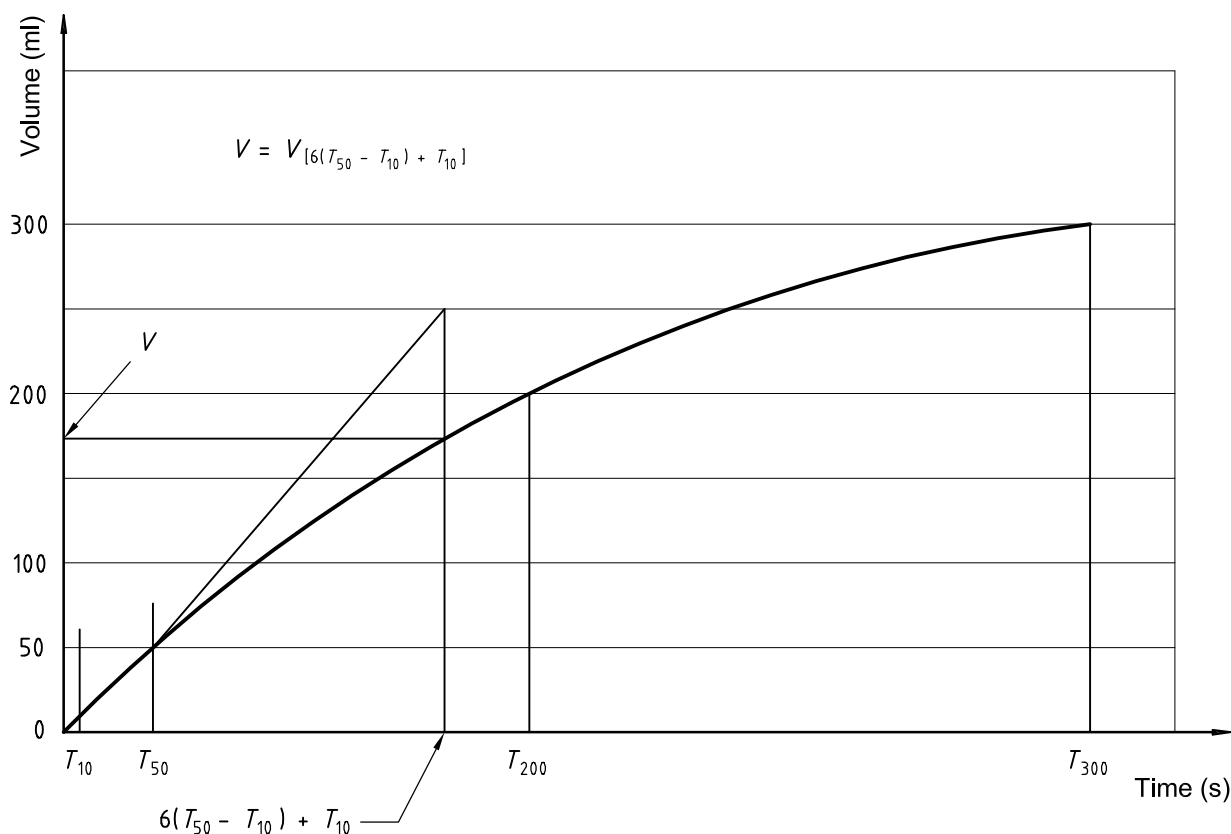


Figure 3 — Diagram of typical filterability test run

9.4 Measure 330 ml \pm 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 \pm 0,02 ml of water (5.1).

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

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 \pm 5 min.

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

9.8 Return the bottle, with the cap slightly loosened, to the oven for 70 h \pm 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 \pm 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 filter membrane 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 (see the note below), in place. Ensure that the apparatus is properly grounded (earthed), that there are no leaks in the pressure system, and that the measuring cylinder (6.3) is properly located below the filtration vessel.

NOTE The correct orientation of the membrane filters is that in which the top is viewed on the normal opening of the packaging box.

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. 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 $60 \text{ s} \pm 10 \text{ s}$. 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 Immediately 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 Inspect the membrane filter for homogeneity of coloration. Repeat the determination if the colour of the membrane filter is significantly uneven.

NOTE An uneven colour indicates that not all of the membrane surface has been 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_1 , using the following equation:

$$F_1 = \frac{V - 10}{240} \times 100$$

where

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

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

10.2 Stage II filterability

Calculate the Stage II filterability, F_{II} , using the following equation:

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

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. Interlaboratory results on the procedure described in this part of ISO 13357 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 can be used as a guide.

13 Test report

The test report shall contain at least the following information:

- a) a reference to this part of ISO 13357;
- 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 the 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 within 5 mm of the bottom of the cylinder (6.3). Capacity approximately 20 ml.

A.1.3 Oven or furnace, controlled to 500 °C, or to the temperature stated in the instructions for the application of the decals (A.1.5).

A.1.4 Balance, capable of weighing up to 500 g with an accuracy of $\pm 0,1$ g.

A.1.5 Decals, ceramic for firing onto glassware.

NOTE Suitable decals are available from Jencons (Scientific) Ltd, Luton, UK. This information is given for the convenience of users of this part of ISO 13357 and does not constitute an endorsement by ISO of these products.

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 $40\text{ g} \pm 0,1\text{ g}$ has been removed. Allow the water to drain down to its new level.

A.2.4 Apply the decal (A.1.5) so that the middle of the mark is in line with the bottom of the meniscus, and that the mark is level.

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 $50\text{ g} \pm 0,1\text{ g}$ has been added. Avoid wetting the cylinder above the final water level during this operation.

A.2.7 Repeat A.2.4 at this new level.

A.2.8 Empty the cylinder and heat in the furnace or oven (A.1.3) to render the marks permanent.

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