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Petroleum products — Determination of the filterability of lubricating oils —

Part 2:
Procedure for dry oils

Produits pétroliers — Détermination de la filtrabilité des huiles lubrifiantes —

Partie 2: Méthode pour les huiles non polluées par de l'eau

Reference number
ISO 13357-2:2005(E)

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Contents

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13357-2 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 13357-2:1998), of which it is a minor revision.

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*

Introduction

The fluid in a hydraulic system acts as a lubricant, and 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, and 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 in a dry state. 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, 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 practical reason why it should not be used with oils of higher viscosity grades, 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 2:
Procedure for dry oils

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 dry lubricating oils, particularly those designed for hydraulic applications. The procedure only applies to mineral-based oils, since fluids manufactured from other materials (e.g. fire-resistant fluids) might 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 dry oils. It is necessary to note that some oils exhibit poorer filterability characteristics in the presence of contaminating water. ISO 13357-1 applies to the investigation of the effect of water and high temperature on filterability, if an oil is used in applications where the presence of water in the oil is likely.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1219-1, *Fluid power systems and components — Graphic symbols and circuit diagrams — Part 1: Graphic symbols*

ISO 3170:2004, *Petroleum liquid — Manual sampling*

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

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 4788:2005, *Laboratory glassware — Graduated measuring cylinders*

ISO 13357-1:2002, *Petroleum products — Determination of the filterability of lubricating oils — Part 1: Procedure for oils in the presence of water*

3 Terms and definitions

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

3.1
filterability
dimensionless number, expressed as a percentage, which is the ratio between two filtration rates

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 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

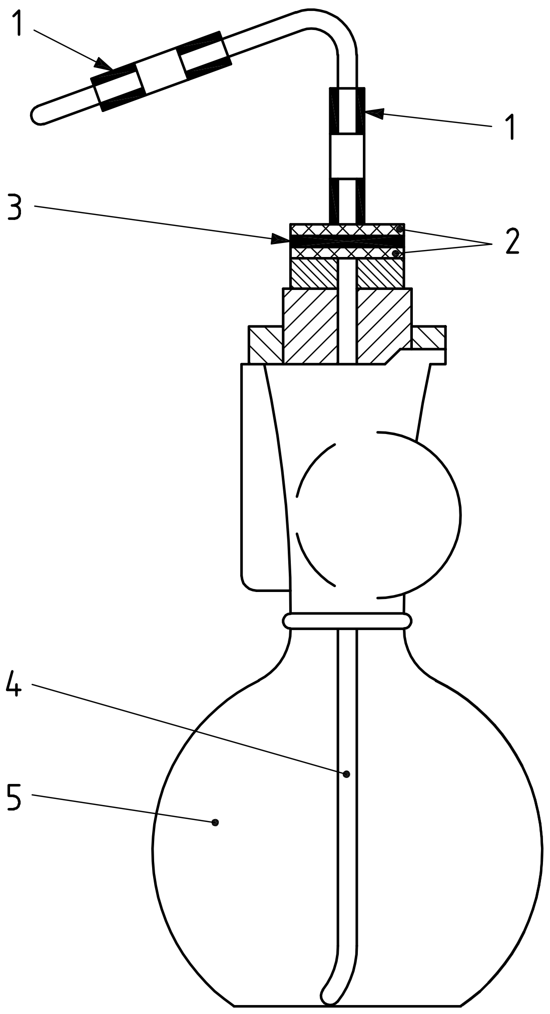
5.1 Water, conforming to grade 3 of ISO 3696:1987.

5.2 Propan-2-ol, 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 in 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.



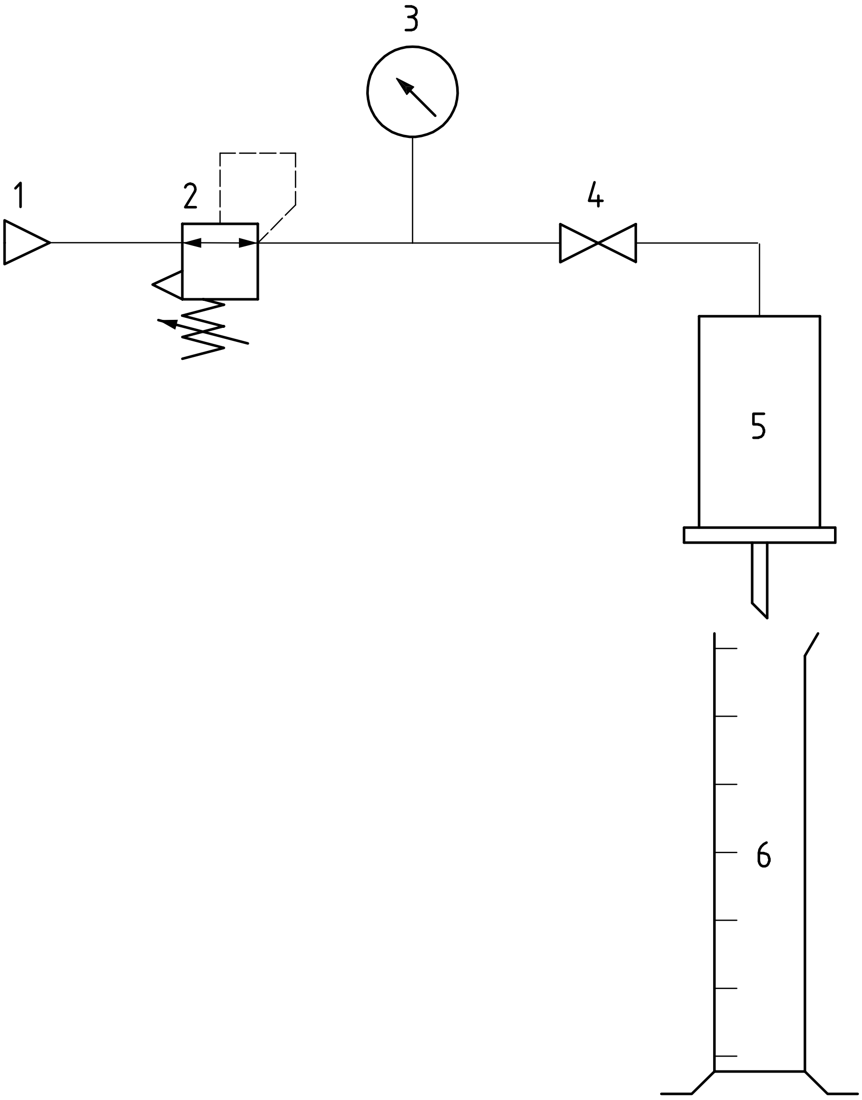
Key

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 the graphic symbols conforming to ISO 1219-1, is shown in Figure 2.



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

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.

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

NOTE Membranes of an equivalent specification to Millipore filter membranes, catalogue number AAWP 047 IF1), have been found satisfactory.

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 320 ml ± 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 9.4) ± 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 ± 10 °C.

6.8 Petri dishes, loosely covered.

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.

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.

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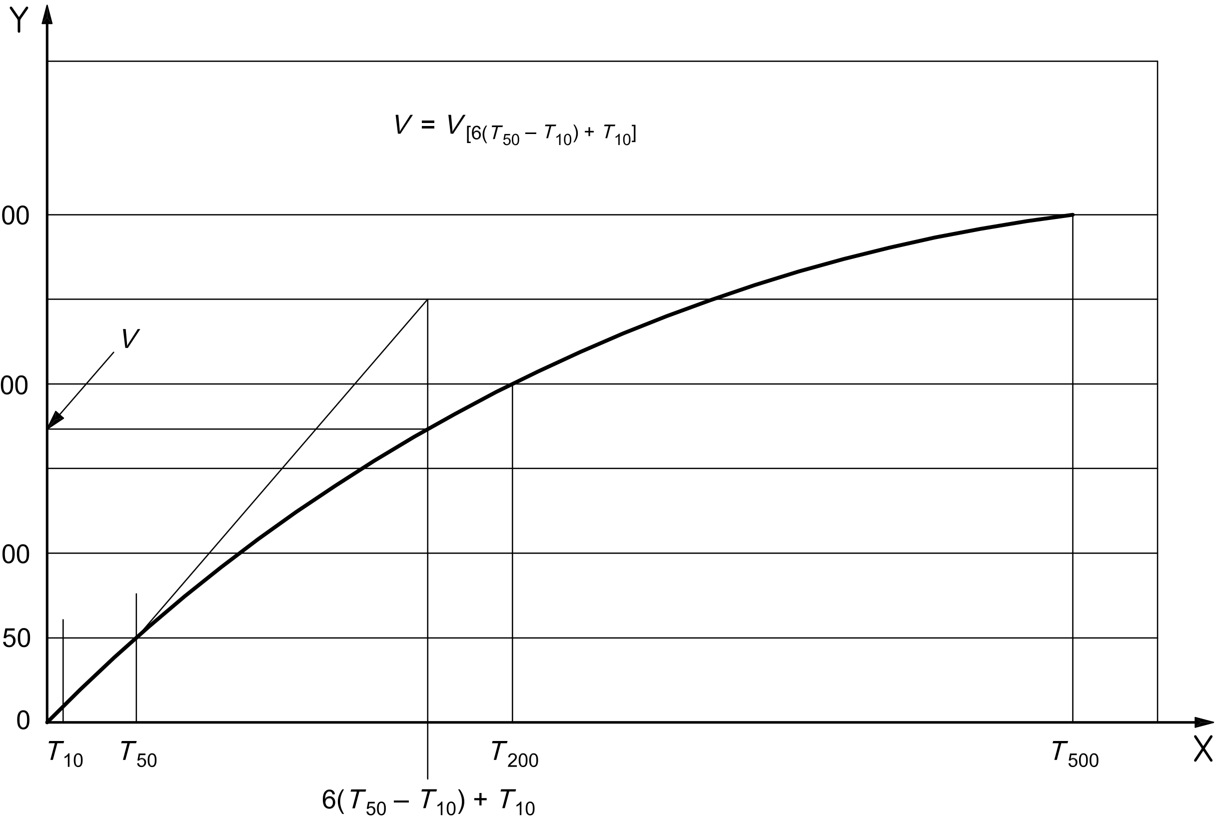
1) Millipore membrane, catalogue number AAWP 047 IF, is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 13357 and does not constitute an endorsement by ISO of this product.

9 Procedure

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



Key

X time, s

Y volume, ml

Figure 3 — Diagram of typical filterability test run

9.1 Carry out the test in triplicate.

9.2 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.

9.3 Assemble the filtration apparatus (6.1) with a membrane filter the correct way up (see Note below), in place. 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.

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

9.4 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

9.5 Mix the laboratory sample by inverting the sample container sharply 30 times in 60 s ± 10 s.

NOTE Each inversion should be completed by a distinct snap.

9.6 Immediately pour 320 ml ± 5 ml of sample into the filtration funnel and close and seal the lid. Open the ball valve and check immediately that the correct pressure is maintained.

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

9.8 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}).

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

9.10 Record the volume in the measuring cylinder at the time (T_V) calculated in 9.9.

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

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

9.13 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.

9.14 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 the equation:

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

where

V is the volume collected at T_V (see 9.10), in millilitres;

T_V is $6(T_{50} - T_{10}) + T_{10}$ (see 9.9), in seconds.

10.2 Stage II filterability

Calculate the Stage II filterability, F_{II}, from the equation:

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

11 Expression of results

11.1 If the three determined values agree within the determinability limit (see 12.1) in all three pairings, report the average of the three determinations, to the nearest 0,1, as the respective filterability, in the form

“Filterability (Stage X) AB,C”

NOTE A, B and C represent numerical figures. A typical result would be “76,2”.

11.2 If one of the three determined values is outside the determinability limit, report the average of the other two determinations in the form

“Filterability (Stage X) (R) AB,C”

Alternatively, perform one more determination, and if this agrees within the determinability limit of the two others, discard the divergent result.

11.3 If all three determined values are outside the determinability limit, discard the result and repeat the test.

11.4 Report the result as “unfilterable” if the filtration time exceeds 7 200 s (2 h).

12 Precision

The precision evaluation programme, performed in 1995, on a matrix of three samples and 20 laboratories, did not conform to the requirements of ISO 4259, and thus only an estimate of precision based on interlaboratory test results is given in 12.1 to 12.3. Table 1 gives estimated values for a range of the average of the results being compared.

12.1 Determinability

The difference between successive determined values, obtained by the same operator with the same apparatus for a series of operations that lead to a single test result would, in the normal and correct operation of the test method, exceed the values below only in one case in 20.

$$d\ (\text{Stage I}) = 0{,}277\sqrt{x(100 - x)}$$

$$d\ (\text{Stage II}) = 0{,}145\sqrt{x(100 - x)}$$

where x is the average of the results being compared.

12.2 Repeatability

The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values below only in one case in 20.

$$r\ (\text{Stage I}) = 0{,}160\sqrt{x(100 - x)}$$

$$r\ (\text{Stage II}) = 0{,}084\sqrt{x(100 - x)}$$

where x is the average of the results being compared.

12.3 Reproducibility

The difference between two test results, independently obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values below only in one case in 20.

$$R\ (\text{Stage I}) = 0{,}396\sqrt{x(100-x)}$$

$$R\ (\text{Stage II}) = 0{,}831\sqrt{x(100-x)}$$

where x is the average of the results being compared.

Table 1 — Estimated precision values

Average test result	Stage I			Stage II		
	d	*r*	*R*	*d*	*r*	*R*
10	8,3	4,8	11,8	4,3	2,5	24,9
30	12,7	7,3	18,1	6,6	3,8	38,0
50	13,8	8,0	19,8	7,2	4,2	43,5
70	12,7	7,3	18,1	6,6	3,8	38,0
90	8,3	4,8	11,8	4,3	2,5	24,9

13 Test report

The test report shall contain at least the following information:

a) reference to this part of ISO 13357;

b) type and complete identification of the product tested;

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

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

e) 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 nearly to the bottom of the cylinder (6.3) with a capacity of approximately 20 ml.

A.1.3 **Oven** or **furnace**, controlled to 500 °C, or the temperature stated by the decal manufacturer.

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

A.1.5 **Decals**[2], 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 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 50,0 g ± 0,1 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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[2] 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.

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