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INTERNATIONAL STANDARD

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Raw optical glass — Resistance to attack by aqueous alkaline solutions at 50 °C — Test method and classification

Verre d'optique brut — Résistance à l'attaque par des solutions aqueuses alcalines à 50 °C *— Méthode d'essai et classification*

Reference number
ISO 10629:1996(E)

Foreword

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International Standard ISO 10629 was prepared by Technical Committee ISO/TC 172, *Optics and optical instruments,* Subcommittee SC 3, *Optical materials and components.*

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Raw optical glass — Resistance to attack by aqueous alkaline solutions at 50 °C — Test method and classification

1 Scope

This International Standard specifies a method for testing the resistance of raw optical glasses to attack by aqueous alkaline solutions at 50 °C and a classification of optical glasses according to the aqueous alkaline resistance (alkali resistance) determined by this method.

This International Standard is applicable to samples of raw optical glasses.

NOTE 1 The test method may also be used for other types of glasses.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2768-1:1989, *General tolerances — Part 1: Tolerances for linear and angular dimensions without individual tolerance indications.*

ISO 3585:1991, *Borosilicate glass 3.3 — Properties.*

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

3 Principle

Attack on polished glass by a test solution (sodium hydroxide 0,01 mol/l solution) at 50 °C for specified times. Weighing to determine the loss in mass and calculation of depth of attack based on the density of the glass. Comparison of the time required for an apparent attack to a depth of 0,1 µm with time scales given in a classification table to obtain the alkali resistance class.

4 Reagents

During the test, unless otherwise stated, use only reagents of recognized analytical grade.

4.1 Water, complying with the grade 2 requirements of ISO 3696.

4.2 Test solution.

Sodium hydroxide solution of concentration c(NaOH) = 0,01 mol/l obtained by diluting the sodium hydroxide solution (4.4) with water. Prepare a carbonate-free solution. Control the pH-value and adjust, if necessary, to pH 12.

4.3 Nitric acid (HNO_3), analytical grade.

Dilute to pH 4,5 ± 0,1.

4.4 Sodium hydroxide solution, c(NaOH) ≈ 0,1 mol/l.

4.5 2-Propanol (C_3H_7OH)

After evaporation of 100 ml of the alcohol, no residue shall be visible. If this is not the case, re-distill 2-propanol.

5 Apparatus

Usual laboratory equipment, and

5.1 Test vessel, cylindrical with a flat base, made of stainless steel, having an internal diameter of 150 mm, a height of 200 mm and a close-fitting lid (see figure 2). The lid has a wide neck and is equipped on the bottom with two hooks from which to suspend the samples. The neck is fitted with a bung of suitable inert material which has previously been boiled in sodium hydroxide solution (4.4) for 60 min, and through which a stirrer can be inserted. Where a gasket is required to ensure an adequate joint between the lid and the body of the vessel, it shall be made of a material which remains inert under the test conditions.

5.2 Stirrer, about 350 mm long, having a 10 mm diameter stainless steel shaft, or a 15 mm diameter polytetrafluoroethylene (PTFE) shaft (see figure 3).

5.3 Platinum or **silver wires,** less than 0,1 mm in diameter, or **cages,** of the same material to receive one sample.

5.4 Heating bath, gas or electrically heated, with a capacity of 30 l to 40 l, thermostatically controlled to maintain the temperature of 50,0 °C ± 0,2 °C.

5.5 Analytical balance, accurate to ± 0,1 mg or better.

5.6 Desiccator, using a 2:1 mixture of silica gel (for H_2O absorption) and soda-lime (for CO_2 absorption) with indicator for regeneration.

5.7 Tongs, protected with inert smooth material, for example plastics.

5.8 Measuring instruments, suitable for measuring lengths and diameter to an accuracy of ± 1 %.

5.9 Ultrasonic equipment for laboratory use, filled with water, which can be heated to at least 50 °C.

5.10 Beakers, made of borosilicate glass 3.3 complying with the requirements of ISO 3585, having a capacity of 100 ml and 250 ml.

5.11 pH-measuring electrode.

6 Preparation of the samples

6.1 General

Cut pieces of the annealed glass (see ISO 9802) to be tested so that after the polishing has been completed the dimensions are nominally 30 mm × 30 mm × 2 mm. Apply the following polishing procedure to all surfaces of the samples using slurry made with water (4.1).

6.1.1 Fine grinding

The fine grinding shall be achieved by using loose abrasive alumina or silicon carbide with the following grain size distribution by mass:

— grains larger than 10,5 µm: ≈ 50 %

— grains larger than 15 µm: < 5 %

— grains larger than 18 µm: None

6.1.2 Polishing

The polishing shall be achieved by using cerium(IV) oxide abrasive having grains smaller than 2 µm and polyurethane LP 26 polisher. The rotation speed for the tool shall be between 50 r/min and 250 r/min and the rotation speed for the sample shall be between 20 r/min and 100 r/min. The pressure (for polishing, not for flatness) shall be between 10 kPa and 40 kPa. The polishing time shall be less than 30 min.

Flatten the sharp edges by a slight polishing (chamfer).

Store the samples in the desiccator (5.6) until they are needed for further processing.

NOTE 2 Soda-lime may attack the glass surface. Great care should be exercised in removing the desiccator lid so as not to disturb any dust.

6.2 Calculation of total surface area

Measure all dimensions to the nearest 0,2 mm and calculate the actual total surface area to an accuracy of 2 %.

NOTE 3 For this purpose, take linear measurements to an accuracy of ± 1 %.

Record the value obtained.

6.3 Cleaning

Samples shall be cleaned as soon as possible after polishing. For this purpose, place three 100 ml beakers (5.10) in the ultrasonic water bath (5.9), containing water heated to 45 °C ± 3 °C. Each beaker shall contain sufficient 2-propanol (4.5) to cover completely any samples which are to be cleaned.

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During the whole cleaning procedure, samples shall be held and transferred by means of tongs (5.7) to avoid surface contamination, such as finger prints.

Immerse the sample in the first beaker for 1 min with the ultrasonic effect applied; then clean the glass with a lightly applied tissue or smooth cloth moistened with 2-propanol. Complete the cleaning by immersing the sample in turn in the second and third beakers, for 1 min in each, with the ultrasonic effect being applied continuously.

Dry the sample by moving it in air and store immediately in the desiccator.

NOTE 4 For drying, a drying oven may also be used for 30 min at 115 °C ± 5 °C.

The 2-propanol in the first beaker shall be replaced after each sample has been cleaned. The 2-propanol in the other beakers shall not be used for more than 10 samples and shall be changed in the event of any suspected contamination.

7 Procedure

7.1 General

The prepared samples shall only be used once.

For the calculation of alkali resistance, at least two samples shall be tested under the same conditions.

Place the test vessel (5.1), filled with 2 l of test solution (4.2) in the heating bath (5.4), adjust the stirrer (5.2) so that it is 15 mm above the vessel bottom and allow the temperature to reach 50,0 °C ± 0,2 °C.

Transfer the cleaned samples, which have been cooled to room temperature in the desiccator, to the analytical balance (5.5) using the tongs (5.7). Weigh and record the mass m_1 of the samples, to an accuracy of ± 0,1 mg. Always use two samples of the same glass for one test in the same test vessel.

Entwine the platinum or silver wire (5.3) crosswise around the samples or put them into the cage (5.3) and hang them so that they are positioned midway between the stirrer shaft and the wall of the test vessel. The underside of the sample shall be 50 mm above the bottom of the test vessel (the whole apparatus is shown in figure 2). There shall be no contact between the sample and the equipment.

Stir with a frequency of 100 r/min.

Reaction times shall be counted from the moment the samples are immersed in the test solution.

After the attack time is completed, remove the samples from the liquid and clean them by the following procedure. Dip each sample for approximately 1 s into each of a series of beakers of capacity 250 ml (5.10) and containing 200 ml of the following liquids. Use the following sequence without pausing:

a) five times into the same water (4.1) at 50 °C. Use fresh water for the next sample;

b) once into nitric acid (4.3) at room temperature;

c) once into water (4.1) at room temperature;

d) three times into 2-propanol (4.5) at room temperature.

Remove the platinum or silver wire (5.3) or the cage (5.3) by holding the sample with the tongs (5.7), immerse it a last time into 2-propanol (4.5) and dry by moving in air (see also note 4 in 6.3). Transfer the clean sample in the desiccator to cool to room temperature. Weigh as soon as possible and record the mass m_2 (after test) to an accuracy of ± 0,1 mg. Calculate the time necessary for an attacked depth of 0,1 µm in accordance with the formula given in clause 8 and observe the changes in the glass surface (see clauses 8 and 9).

NOTE 5 For this purpose, observe the glass surface under natural light or under illlumination by a microscope lamp at an angle of approximately 45°.

7.2 Testing unknown glasses

The following preliminary measurements for the determination of the time of attack are necessary.

Prepare six samples in accordance with clause 6 and test, only one sample at a time, following the sequence given in figure 1.

Start the test by immersing one sample into the test solution (4.2) for 1 h. Depending on the loss in mass, calculate for classification or continue with the next attack (4 h or 0,25 h, see sequence in figure 1). Normally the class is calculated when the loss in mass is between 1 mg/sample and 4 mg/sample. When the loss in mass is less than 1 mg/sample after the 4 h attack, apply a further attack for 16 h. The result after this time of attack is to be used for calculation in every case as well as the result obtained when the loss in mass is more than 1 mg/sample after the 4 h attack.

When the time of attack is determined, continue in accordance with 7.3.

7.3 Testing known glasses

If the alkali resistance class of an optical glass is reasonably well known or determined in accordance with 7.2, the following procedure shall be applied.

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Immerse two samples in the same test solution for the expected time, i.e. 0,25 h, 1 h or 4 h. If the loss in mass is less than 1 mg/sample after 4 h, apply a further test for 16 h.

If the loss in mass is clearly less than 1 mg/sample, proceed with the next longer attack. If the loss in mass is clearly more than 4 mg/sample after 1 h, proceed with the next shorter attack (see figure 1).

The results from attack with loss in mass between 1 mg/sample and 4 mg/sample after 1 h, or more than 1 mg/sample after 0,25 h or 4 h, or from attack after 16 h, shall be used for the calculation of the alkali resistance class.

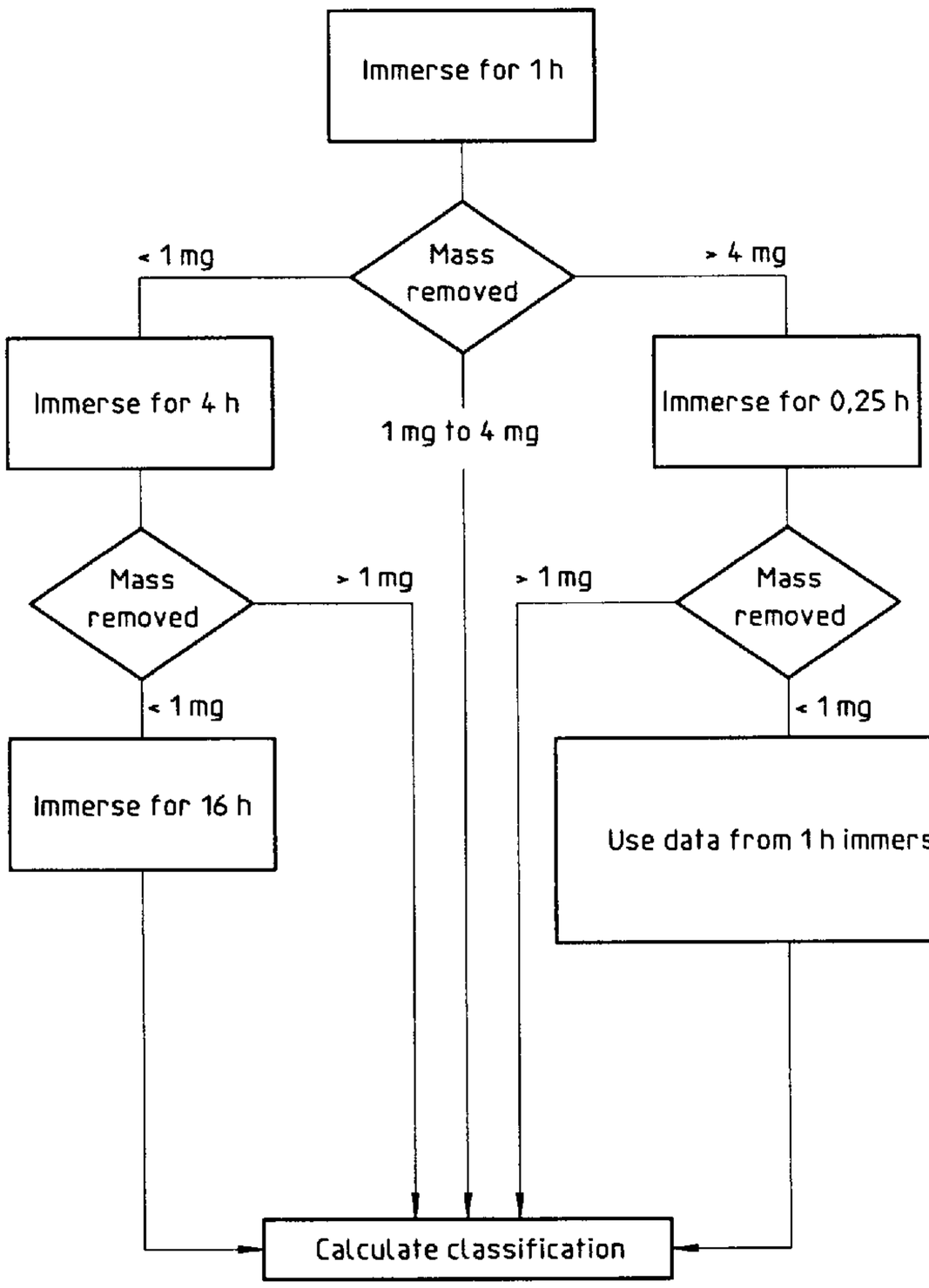

Figure 1 — Sequence of testing the alkali resistance of an unknown optical glass

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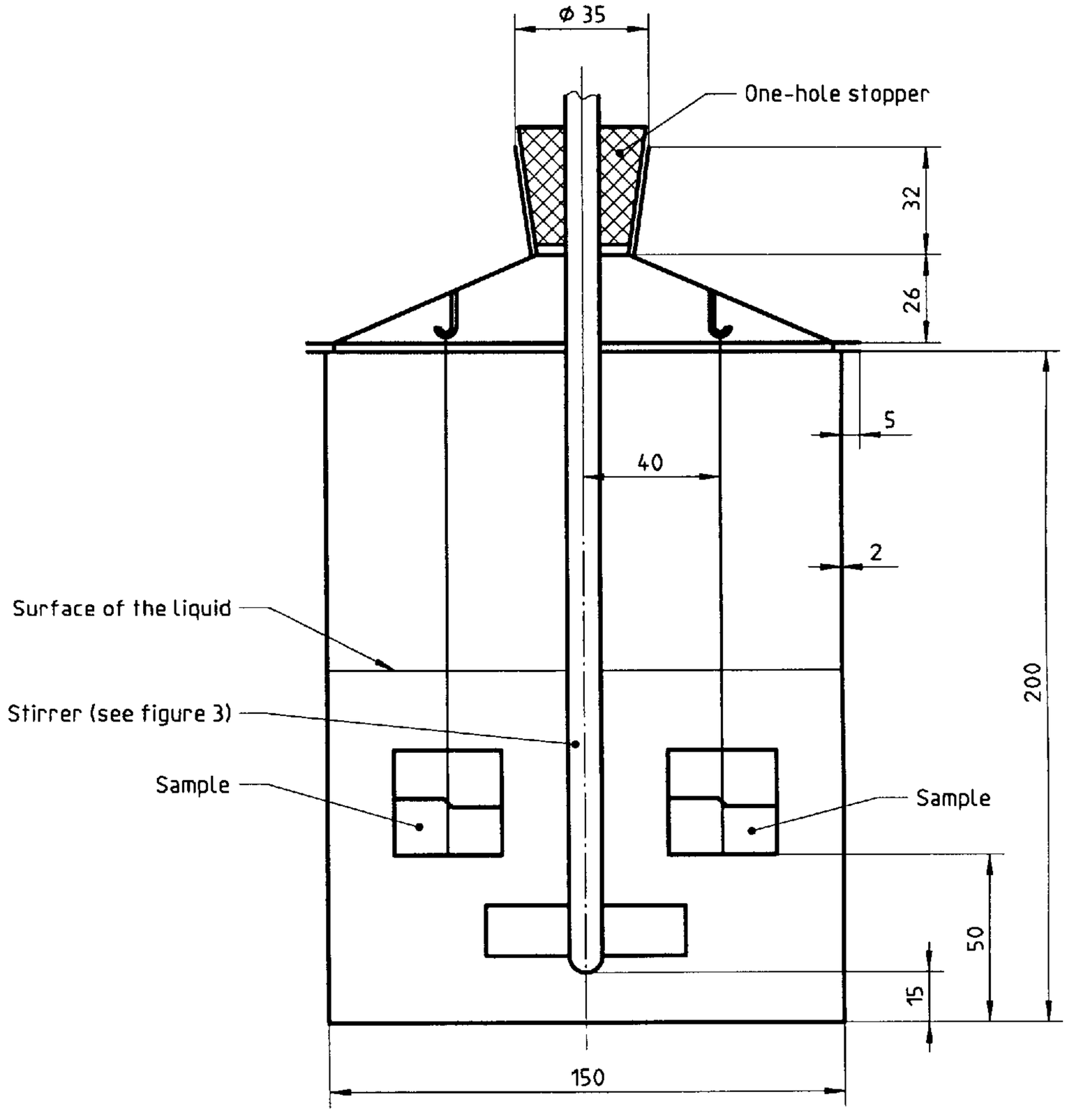
Dimensions in millimetres



Execution:

— 2 hooks soldered to the lid

— 1 flange with ground-flat surface, fixed to lid

NOTE — Permissible variations in dimensions without tolerance indication shall be in accordance with those specified for the coarse series in ISO 2768-1.

Figure 2 — Test apparatus

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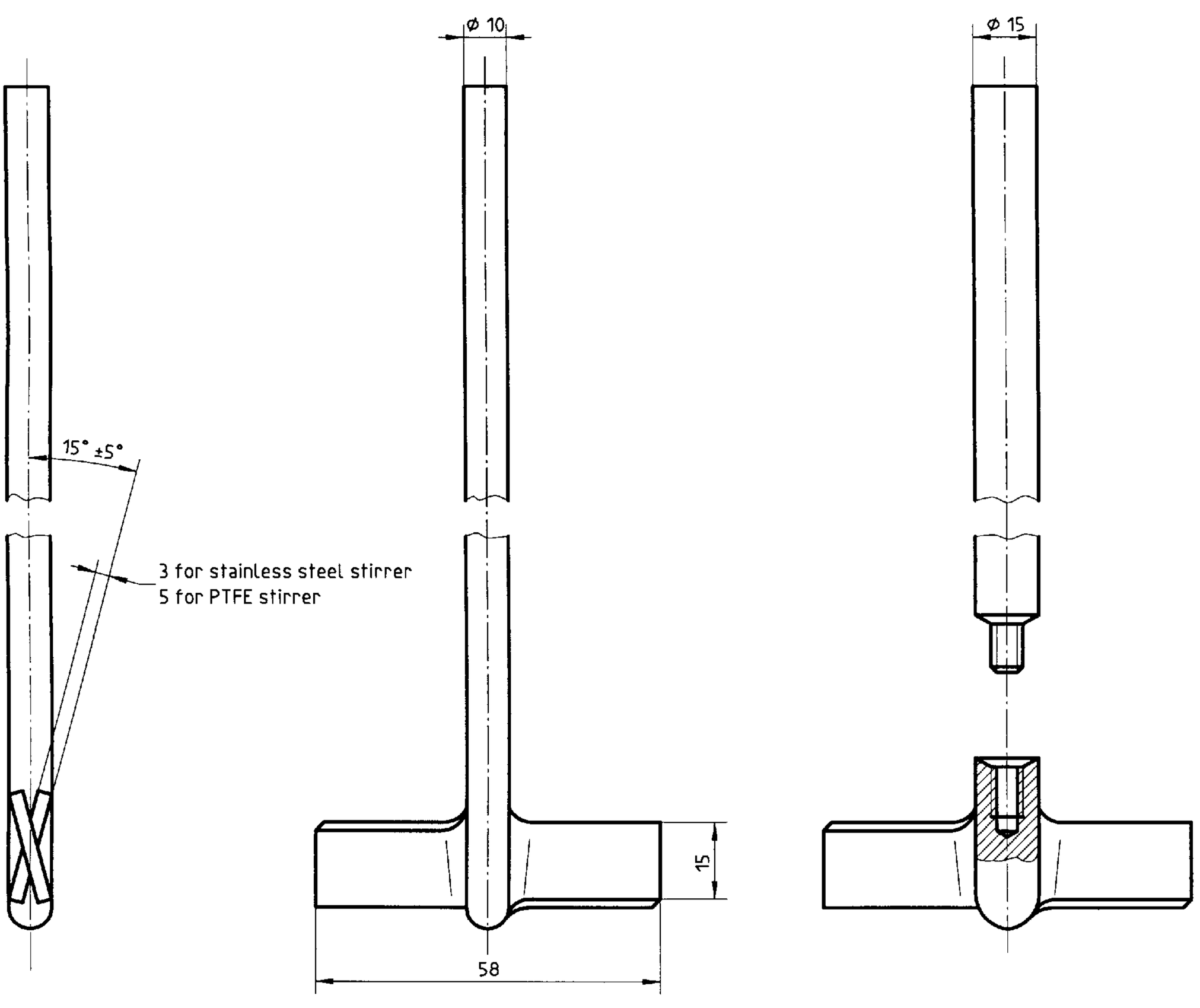
Dimensions in millimetres



a) Stainless steel

b) PTFE

NOTE — Permissible variations in dimensions without tolerance indication shall be in accordance with those specified for the coarse series in ISO 2768-1.

Figure 3 — Stirrers

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8 Expression of results

From the mean value of the loss in mass determined in accordance with 7.3, calculate the time $t_{0,1}$, in hours, necessary to etch a surface layer to a depth of 0,1 µm using the following formula:

$$t_{0,1} = \frac{t_e \times \rho \times A}{(m_1 - m_2) \times 100}$$

where

t_e is the time for attack in the experiment, in hours;

ρ is the density of the glass, in grams per cubic centimetre;

A is the total surface area of the sample, in square centimetres;

m_1 is the mass of the sample before the test, in milligrams;

m_2 is the mass of the sample after the test, in milligrams.

9 Classification and designation

Optical glasses shall be classified in accordance with table 1, according to the time $t_{0,1}$ in hours, necessary to etch a surface layer to a depth of 0,1 µm, when tested by the method specified in this International Standard.

Changes in the surface of the sample which are visible after determining the mass m_2 (see clause 7) used for the calculation (see clause 8) are qualitatively evaluated with the naked eye and given in addition to the class number as follows:

.0: no visible changes

.1: clear, but irregular surface (wavy, pockmarked, pitted)

.2: staining and/or interference colours (slight selective leaching)

.3: tenacious thin whitish layer (stronger selective leaching, a cloudy/hazy/dullish surface)

.4: loosely adhering thick layer, such as insoluble, friable surface deposit (may be a cracked and/or peelable surface, surface crust, or cracked surface; strong attack)

Table 1 — Classification of optical glasses

Alkali resistance class AR	Time needed to etch to a depth of 0,1 µm, $t_{0,1}$ h
1	> 4
2	from 4 to 1
3	from < 1 to 0,25
4	< 0,25

Differences in the history of glass or in its pretreatment during fine grinding or polishing (see 6.1) may be responsible for a deviation of one place in the additional numbers to the class.

For convenience of reference to the alkali resistance of optical glass complying with the classification laid down in this International Standard, the designation shown in the following example shall be used.

EXAMPLE

For a glass having a density $\rho = 3{,}31$ g/cm^3, a total surface area $A = 20{,}4$ cm^2, a loss in mass $(m_1 - m_2) = 3{,}7$ mg/sample, after an attack time $t_e = 1$ h, resulting in $t_{0,1} = 0{,}18$ h for the attack to a depth of 0,1 µm and with interference colours visible after the attack:

Optical glass, alkali resistance class ISO 10629-AR 4.2

10 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) identification of the samples, including density;

c) the surface area tested, in square centimetres;

d) statement as to which attack time gave the results for the calculation of the time for the attack of the surface layer to a depth of 0,1 µm, and observation of any changes in the surface;

e) the number of samples tested under the final conditions and taken for the mean value;

f) the designation of the alkali resistance class AR;

g) any unusual features noted during the determination.

Annex A
(informative)

Bibliography

[1] ISO 9802: —[1)], *Raw optical glass — Vocabulary.*

1) To be published.

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Descriptors: glass, optical glass, raw materials, chemical resistance, alkali resistance, classification, tests, chemical tests, alkali resistance tests, test equipment, designation.

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