

INTERNATIONAL STANDARD

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Raw optical glass — Resistance to attack by aqueous acidic solutions at 25 °C — Test method and classification

*Verre d'optique brut — Résistance à l'attaque par des solutions acides
aqueuses à 25 °C — Méthode d'essai et classification*



Reference number
ISO 8424:1996(E)

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.

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.

International Standard ISO 8424 was prepared by Technical Committee ISO/TC 172, *Optics and optical instruments*, Subcommittee SC 3, *Optical materials and components*.

This second edition cancels and replaces the first edition (ISO 8424:1987), of which it constitutes a technical revision.

Annex A of this International Standard is for information only.

Raw optical glass — Resistance to attack by aqueous acidic solutions at 25 °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 acidic solutions at 25 °C and a classification of optical glasses according to the acid resistance determined by this method.

This International Standard contains basic information about the chemical durability of the glass tested.

It 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 test solution with a pH of 0,3 (nitric acid solution 0,5 mol/l) or 4,6 (buffer sol-

ution) at 25 °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 acid 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 Nitric acid, solution [$c(\text{HNO}_3) = 0,5 \text{ mol/l}$], pH $0,3 \pm 0,05$.

4.3 Acetic acid (CH_3COOH), $\rho = 1,05 \text{ g/cm}^3$, 100 % (m/m).

4.4 Sodium hydroxide, solution [$c(\text{NaOH}) = 1 \text{ mol/l}$].

Dissolve 40 g of sodium hydroxide in water and dilute to 1 litre.

4.5 2-Propanol, ($\text{C}_3\text{H}_7\text{OH}$).

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

4.6 Buffer solution, pH $4,6 \pm 0,05$.

In a one-mark 1 000 ml volumetric flask, mix 11,8 ml of the acetic acid (4.3), 200 ml of water and 100 ml of the sodium hydroxide solution (4.4). Make up to the mark with water. Store in a plastic or boro-silicate glass bottle.

5 Apparatus

Usual laboratory equipment, and

5.1 Beaker, flat flange, made of borosilicate glass 3.3, complying with the requirements of ISO 3585, having a capacity of 2 000 ml, an internal diameter of 150 mm, an external diameter of 153 mm and a height of 200 mm (see figure 2).

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

5.3 Platinum 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 $25,0\text{ }^{\circ}\text{C} \pm 0,2\text{ }^{\circ}\text{C}$.

5.5 Analytical balance, accurate to $\pm 0,1\text{ mg}$ or better.

5.6 Desiccator, using a 2:1 mixture of silicagel (for H_2O absorption) and soda lime (a mixture of CaO and Na_2O , 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 $\pm 1\text{ }%$.

5.9 Ultrasonic equipment for laboratory use, filled with water, which can be heated to at least $50\text{ }^{\circ}\text{C}$.

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

5.11 pH-measuring electrode.

5.12 Stop watch, range min. 100 min, accuracy $\pm 0,1\text{ }%$.

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 $30\text{ mm} \times 30\text{ mm} \times 2\text{ mm}$. 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 $10,5\text{ }\mu\text{m}$: $\approx 50\text{ }%$
- grains larger than $15\text{ }\mu\text{m}$: $< 5\text{ }%$
- grains larger than $18\text{ }\mu\text{m}$: none

6.1.2 Polishing

The polishing shall be achieved by using cerium(IV) oxide abrasive having grains smaller than $2\text{ }\mu\text{m}$ 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 to 40 kPa. The polishing time shall be less than 30 min.

Flatten the sharp edges by 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 $0,2\text{ mm}$ and calculate the actual total surface area to an accuracy of $2\text{ }%$.

NOTE 3 For this purpose, take linear measurements to an accuracy of $\pm 1\text{ }%$.

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 $45\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$. Each beaker shall contain sufficient 2-propanol (4.5) to cover completely any samples which are to be cleaned.

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

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 $115\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

The 2-propanol in the first beaker shall be replaced after each sample has been cleaned. The isopropyl alcohol 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 be used only once.

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

Place the test beaker (5.1) filled with 2 litres of test solution 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 $25,0\text{ }^{\circ}\text{C} \pm 0,2\text{ }^{\circ}\text{C}$.

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 as m_1 , to an accuracy of $\pm 0,1\text{ mg}$. Always use two samples of the same glass for one test in the same test beaker.

Entwine the platinum 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 rod and the wall of the test beaker. The underside of the sample shall be 50 mm above the bottom of the test beaker (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, remove the samples from the liquid, wash twice with distilled water and remove the platinum wires. Immerse the samples three times 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 as m_2 (after test) to an

accuracy of $\pm 0,1\text{ mg}$. Calculate the time necessary for an attacked depth of $0,1\text{ }\mu\text{m}$ in accordance with the formula given in clause 8 and observe the changes in the glass surface (see clause 8 and clause 9).

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

7.2 Testing unknown glasses

For this purpose, the following preliminary measurements for the determination of the conditions (time and solution) of attack are necessary.

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

Start the test by immersing one sample into the test solution (4.6) for 16 h. Depending on the loss in mass, calculate for classification or continue with the next attack with the same pH for 100 min or pH 0,3 for 16 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. Consequently, for the test under laboratory conditions, there are six possibilities for attack.

When the conditions for attack have been determined, continue according to 7.3 (see table 1).

7.3 Testing known glasses

If the acid resistance class of an optical glass is reasonably well known or determined in accordance with 7.2, the procedure given in 7.1 under the conditions specified in table 1 shall be applied.

The results from attack with loss in mass between 1 mg/sample and 4 mg/sample shall be used for the calculation of the acid resistance class.

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

Table 1 — Different solutions and specified test times for attack of optical glasses in the acid resistance test with approximate acid resistance class SR

Test solution		Test times	Approximate acid resistance class SR*	
No.	pH	min(h)		
(3c)	4,6	10 (0,17)	Weakest attack ↓ Strongest attack	53
(2b)	4,6	100 (1,67)		52
(1)	4,6	(16)		51 or 5
(3b)	0,3	100 (1,67)		4 or 5
(2a)	0,3	(16)		3
(3a)	0,3	(100)		2 or 1

* The correct acid resistance class SR according to table 2 is calculated by the equation given in clause 8 using the loss in mass determined according to 7.3.

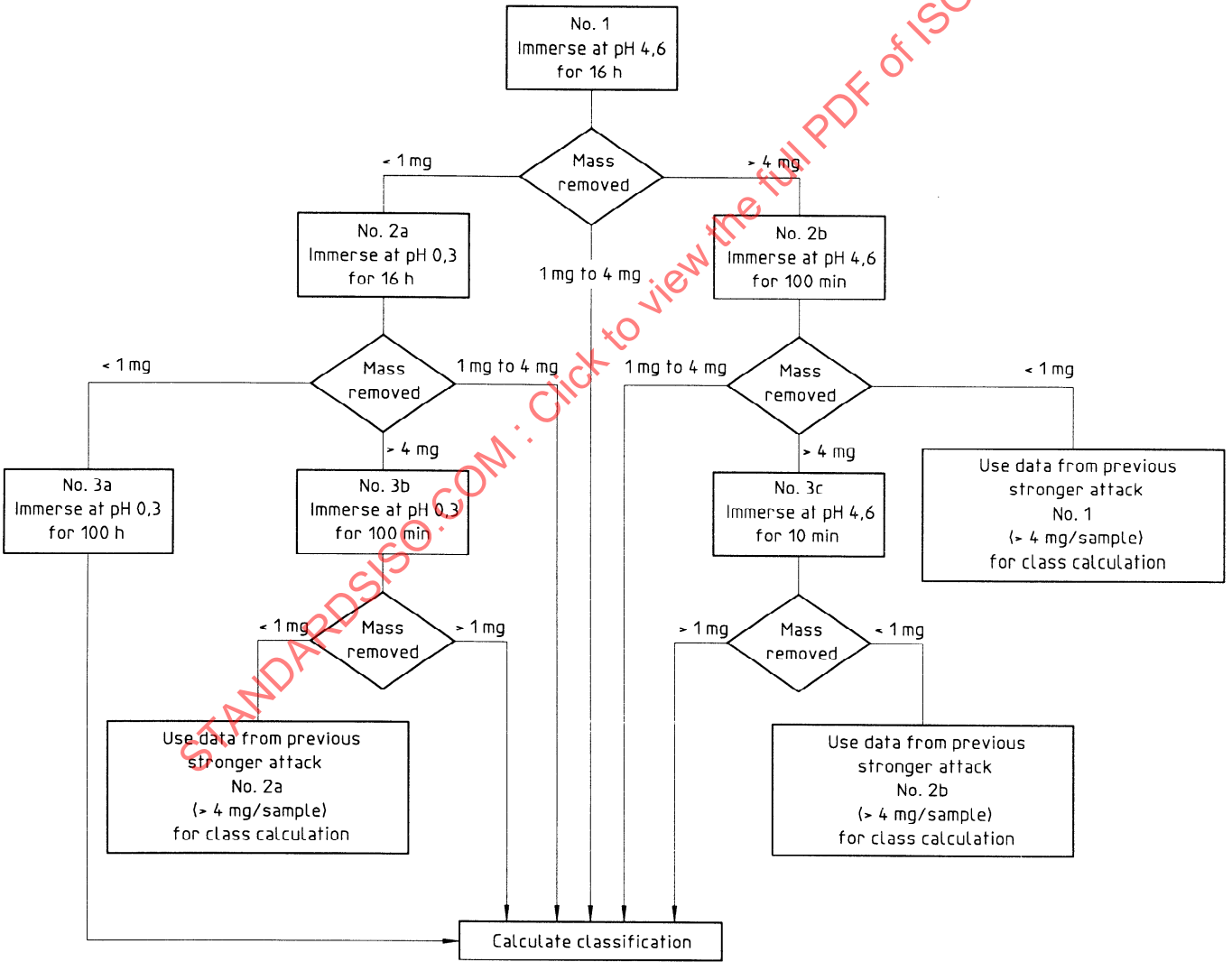


Figure 1 — Sequence of testing the acid resistance of an unknown optical glass

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 2, 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, pock-marked, 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)

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

Table 2 — Classification of optical glasses

Acid resistance class SR	pH of the attacking solution	Time needed to etch to a depth of 0,1 μm , $t_{0,1}$ h
1	0,3	> 100
2	0,3	from 100 to 10
3	0,3	from < 10 to 1
4	0,3	from < 1 to 0,1
5	0,3	< 0,1
	4,6	> 10
51	4,6	from 10 to 1
52	4,6	from < 1 to 0,1
53	4,6	< 0,1

For convenience of reference to the acid 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

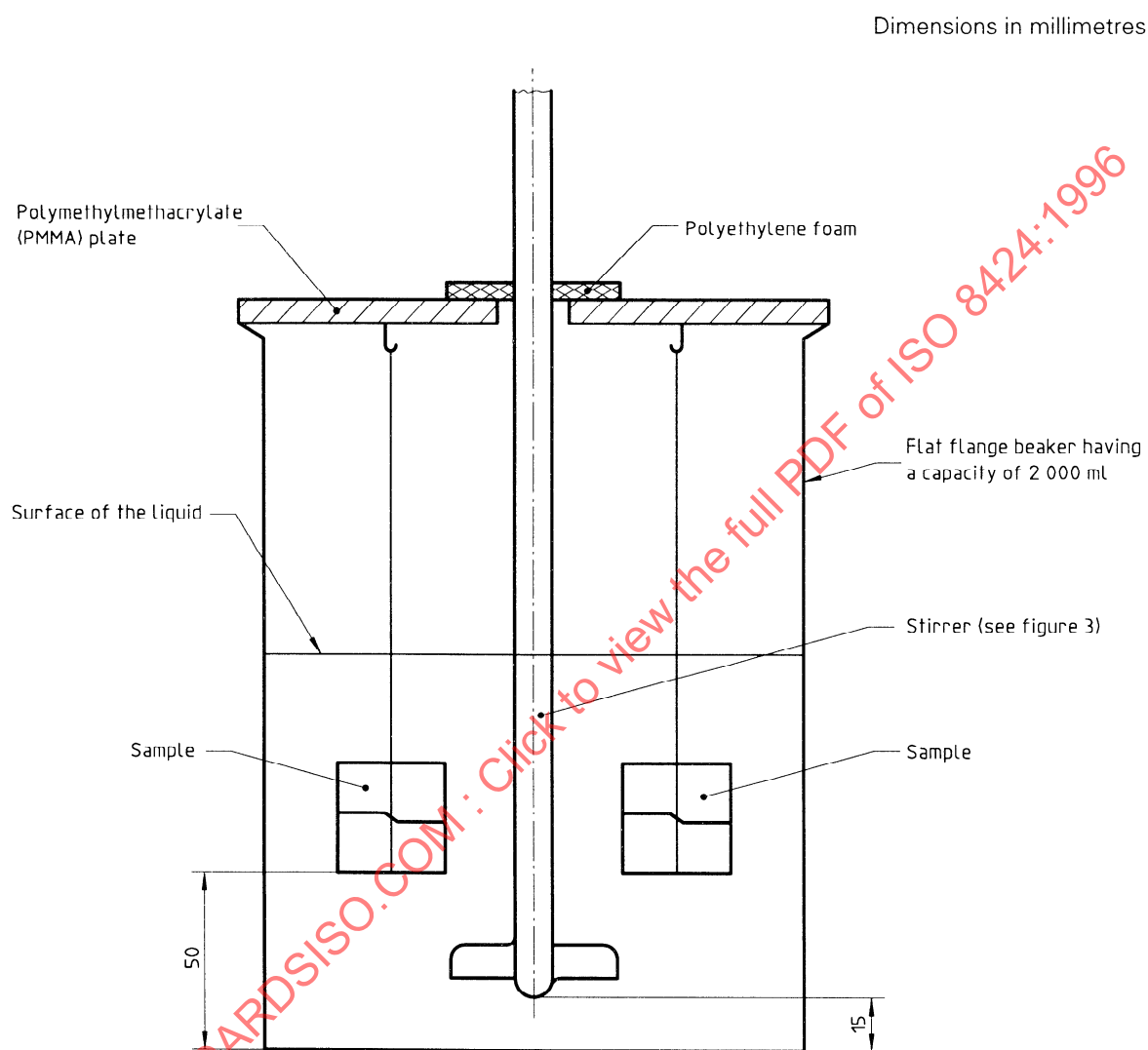
For a glass having a density $\rho = 3,31 \text{ g/cm}^3$, a total surface area $A = 20,4 \text{ cm}^2$, a loss in mass $(m_1 - m_2) = 3,7 \text{ mg/sample}$, after an attack time $t_e = 100 \text{ min} (= 1,67 \text{ h})$ by an attacking solution of pH 0,3, resulting in $t_{0,1} = 0,30 \text{ h}$ for the attack to a depth of 0,1 μm and with interference colours visible after the attack:

Optical glass, acid resistance class ISO 8424-SR 4.2

10 Test report

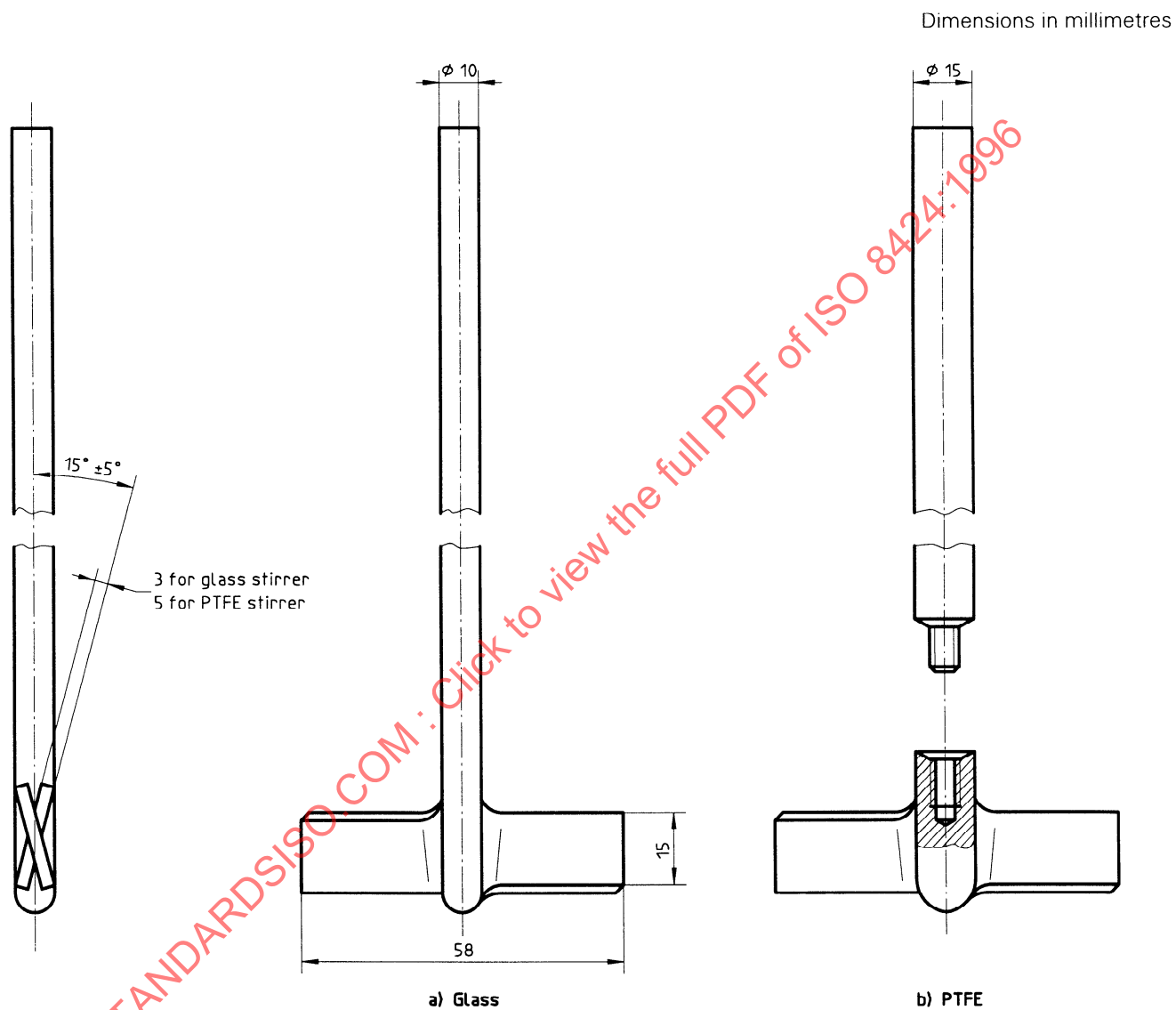
The test report shall include the following information:

- a) a reference to this International Standard;
- b) identification of the samples, including density;
- c) the surface area tested, in square centimetres;
- d) the sequence of steps, carried out in accordance with figure 1, a note stating which step 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 acid resistance class SR;
- g) any unusual features noted during the determination.



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



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