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Raw optical glass — Testing of the resistance to attack by aqueous acidic solutions at 25 °C and classification

Verre d'optique brut — Essai de résistance à l'attaque par des solutions acides aqueuses à 25 °C et classification

Reference number
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Foreword

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International Standard ISO 8424 was prepared by Technical Committee ISO/TC 172, *Optics and optical instruments.*

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Raw optical glass — Testing of the resistance to attack by aqueous acidic solutions at 25 °C and classification

0 Introduction

This International Standard is based on a test method approved by the International Optical Glass Expert Group of Technical Committee 2 "Chemical durability and analysis" of the International Commission on Glass (ICG/TC 2).

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.

2 Field of application

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

3 References

ISO 3585, *Glass plant, pipeline and fittings — Properties of borosilicate glass 3.3.*

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

4 Principle

Attack on polished glass by an aqueous solution with a pH of 0,3 or 4,6 respectively at 25 °C for specified times. Weighing of the loss in mass and calculation of depth of attack based on the density of the glass. Comparison of the time required for the apparent attack to a depth of 0,1 µm with time scales given in the classification table to obtain the acid resistance class.

5 Reagents

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

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

5.2 Nitric acid, c (HNO_3) = 0,5 mol/l (pH 0,3) solution.

5.3 Acetic acid (CH_3COOH) ϱ = 1,05 g/cm³, 100 % (m/m).

5.4 Sodium hydroxide, solution, c (NaOH) = 1 mol/l.

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

5.5 Isopropyl alcohol (C_3H_7OH).

After evaporation of 100 ml of the alcohol, no residue shall be visible. If this is not the case, redistill.

5.6 Buffer solution, pH 4,6

Mix, in a one-mark 1 000 ml volumetric flask, 11,8 ml of the acetic acid solution (5.3), 200 ml of water and 100 ml of the sodium hydroxide solution (5.4). Make up to the mark with water. Store in a plastic or borosilicate glass bottle.

6 Apparatus

Usual laboratory equipment, and

6.1 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).

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

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

6.4 Heating bath, gas or electrically heated, with a 30 to 40 litre capacity, thermostatically controlled to maintain a temperature of 25,0 ± 0,2 °C.

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

6.6 Desiccator, using a 2 : 1 mixture of silicagel and soda lime, crystalized, for carbon dioxide and water absorption.

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

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

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

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

7 Preparation of the samples

7.1 Polishing

Cut pieces of the glass 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 (5.1).

7.1.1 Fine grinding

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

- grains larger than 10,5 µm : ≈ 50 %
- grains larger than 15 µm : < 5 %
- grains larger than 18 µm : None

7.1.2 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 and 250 r/min and the rotation speed for the sample shall be between 20 and 100 r/min. The pressure (for polishing, not for flatness) shall be between 10 and 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 (6.6) until they are needed for further processing.

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

7.2 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 — For this purpose, take linear measurements to an accuracy of ± 1 %.

Record the value obtained.

7.3 Cleaning

Samples shall be cleaned as soon as possible after polishing. For this purpose, place three 100 ml beakers (6.10) in the ultrasonic water bath (6.9), containing water heated to 45 ± 3 °C. Each beaker shall contain sufficient isopropyl alcohol (5.5) to cover well any samples which are to be cleaned.

During the whole cleaning procedure, samples shall be held and transferred by means of tongs (6.7) 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 isopropyl alcohol. 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 — For drying, a drying oven may also be used, for 30 min at 120 °C.

The isopropyl alcohol 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.

8 Procedure

8.1 General

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

Place the test beaker (6.1), filled with 2 litres of test solution in the heating bath (6.4), adjust the stirrer (6.2) so that it is 15 mm above the vessel bottom and allow the temperature to reach 25 ± 0,2 °C.

Transfer the cleaned samples, which have been cooled to room temperature in the desiccator, to the weighing balance (6.5) using the tongs (6.7). Weigh and record the mass as m_1, weighed to an accuracy of ± 0,1 mg. Always use two samples of the same glass for the one test in the same test beaker.

Entwine the platinum wire (6.3) crosswise around the samples or put them into the cage (6.3) 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 is completed, take the samples out of the liquid, wash twice with distilled water and remove the platinum wires. Immerse the samples three times in isopropyl alcohol,

dry by moving in air and store in the desiccator. Weigh as soon as possible and record the mass as m_2 (after test) to an accuracy of ± 0,1 mg. Calculate the time for an attacked depth of 0,1 µm in accordance with the formula given in clause 9 and observe the changes in the glass surface (see clauses 9 and 10).

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

8.2 Testing unknown glasses

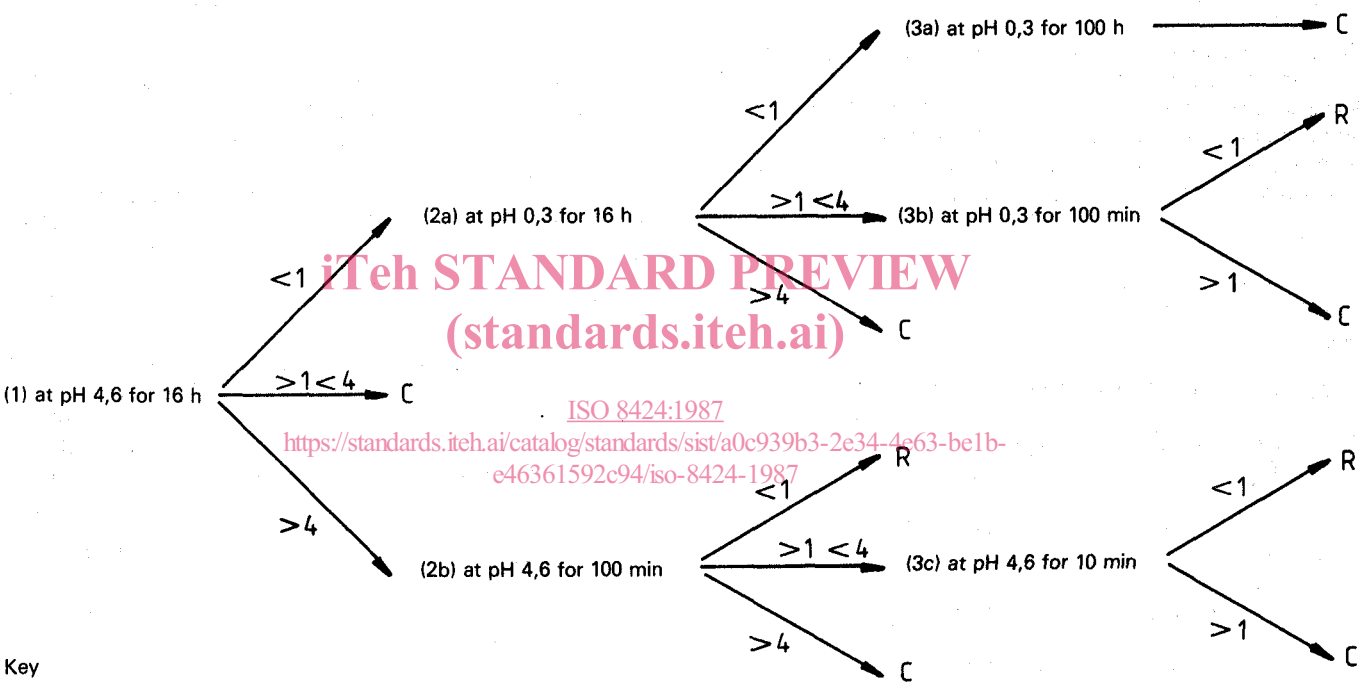
For this purpose the following preliminary measurements for the determination of the conditions (time and solution) of attack are necessary.

Prepare six samples in accordance with clause 7 and test, step by step only one sample at the time, following the sequence given in figure 1.

Start with the buffer solution (5.6) for 16 h. Continue, depending on the loss in mass, with the next solution (i.e. with the same pH or pH 0,3 and the appropriate time (see sequence in figure 1).

For determination of the acid resistance class, use the loss in mass between 1 mg/sample and 4 mg/sample. In principal, follow figure 1.

Consequently, for the test under laboratory conditions, there are six possibilities for attack. When the conditions for attack have been determined continue according to 8.3. (See table 1.)



Key

(1), (2a), (2b), (3a), (3b), (3c) : Number of the test solution

1 or 4 : 1 mg/sample or 4 mg/sample loss in mass after the test

C : Calculate for classification

R : Calculate for classification using the result of the previous stronger attack (> 4 mg/sample)

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

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*	
N°	pH	min (h)		
(3c)	4,6	10 (0,17)	Weakest 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)	Strongest attack	2 or 1

* The correct acid resistance class SR according to table 2 is calculated by the equation given in clause 9 using the loss in mass determined according to 8.3.

8.3 Testing known glasses

If the acid resistance class of an optical glass is reasonably well known, then the procedure given in 8.1 under the conditions specified in table 1 or determined according to 8.2 shall be followed.

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).

9 Expression of results

Calculate the mean value of the loss in mass observed for samples which correspond to the loss in mass between 1 and 4 mg/sample, or to the minimum condition (i.e. test solution and test time, C or R in figure 1). From this value, 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 :

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

where

t_e is the attack time 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.

10 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.

Table 2 — Classification of optical glasses

Acid resistance class SR	pH of the attacking solution	Time, $t_{0,1}$, needed to etch to a depth of 0,1 µm 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

Changes in the surface of the sample which are visible after determining the mass m_2 (see clause 8) used for the calculation (see clause 9) 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 crust, or cracked surface) (strong attack).

Differences in the history of glass or in its pre-treatment during fine grinding or polishing (see 7.1) may be responsible for slight deviations in the additional numbers to the class numbers.

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

Example

For a glass, having a density ϱ = 3,31 g/cm³, a total surface area A = 20,4 cm², a loss in mass ($m_1 - m_2$) = 3,7 mg/sample after an attack time t_e = 100 min (= 1,67 h) by an attacking solution of pH 0,3, resulting in $t_{0,1}$ = 0,30 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

11 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 tested, in square centimetres;

d) the sequence of steps, carried out in accordance with figure 1, a statement as to which step gave the results for the calculation of the time for the attack of 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.

Dimensions in millimetres

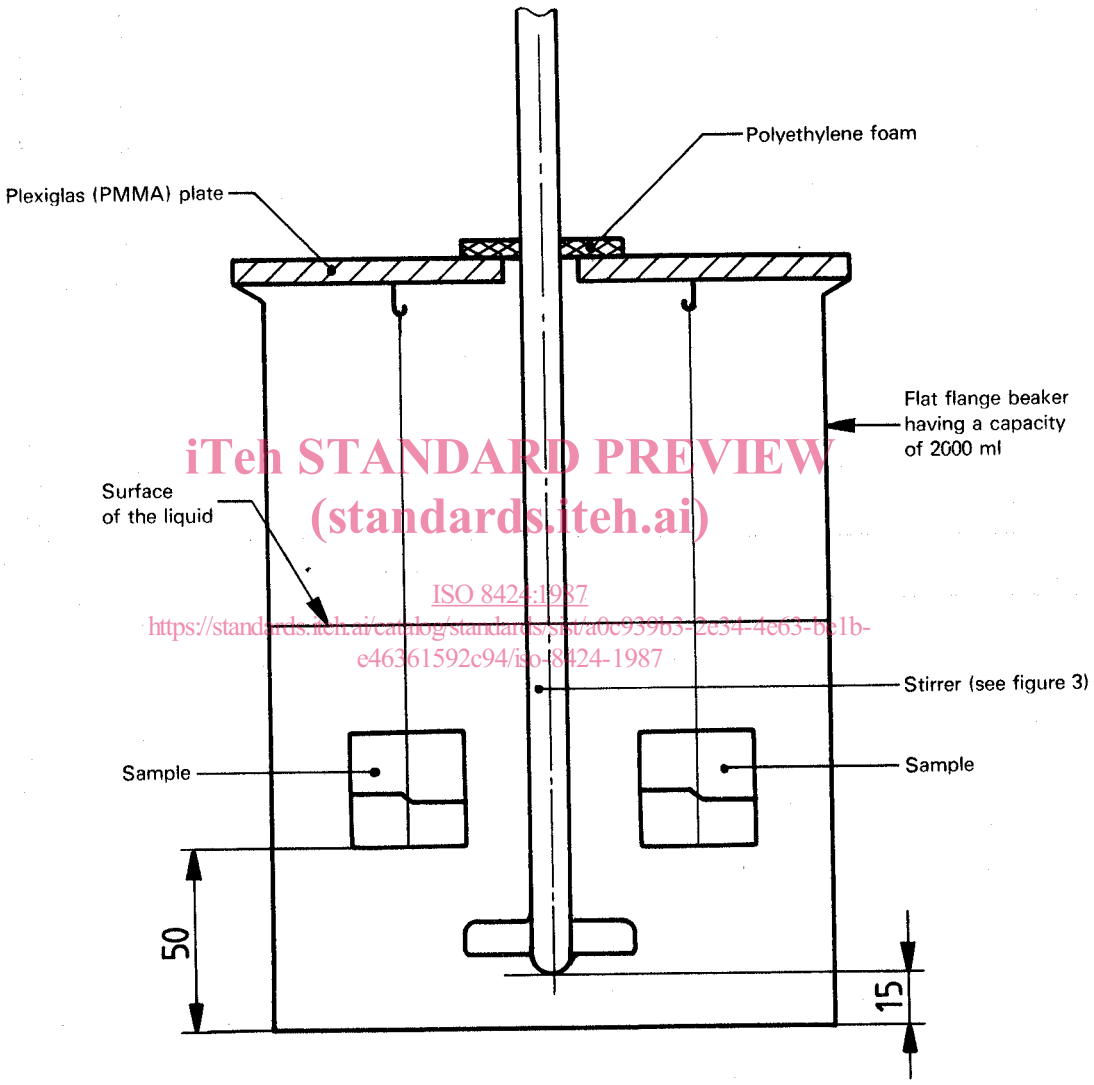


Figure 2 — Test apparatus

Dimensions in millimetres

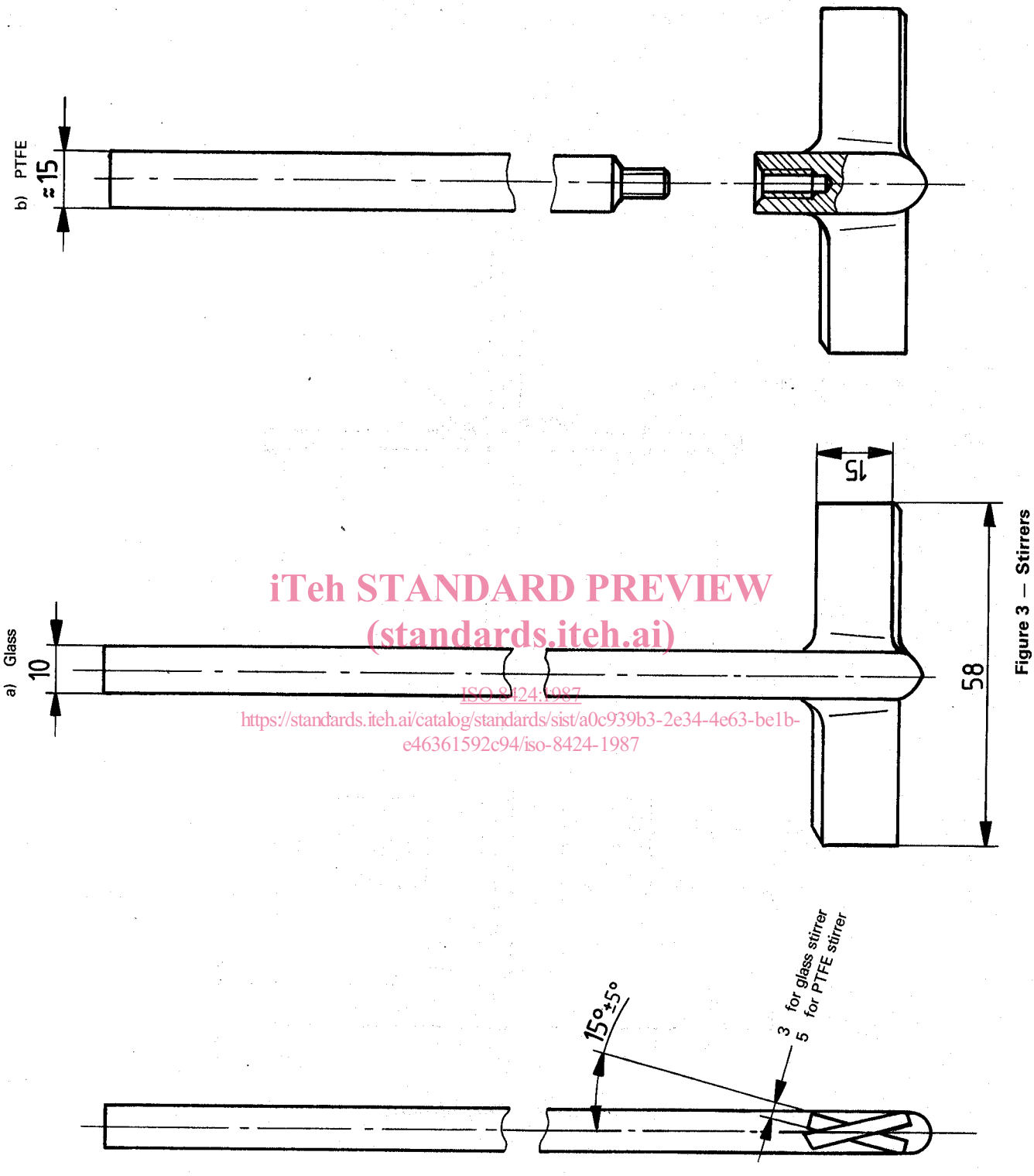


Figure 3 — Stirrers

UDC 666.22 : 620.193.41

Descriptors : optical equipment, optical glass, classification, tests, acid resistance tests.

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