INTERNATIONAL STANDARD

ISO 9689

First edition
1990-12-01

Raw optical glass — Resistance to attack by aqueous alkaline phosphate-containing detergent solutions at 50 °C — Testing and classification

Verre d'optique brut — Résistance à l'attaque par des solutions aqueuses de détergent contenant du phosphate alcalin à 50 °C — Essai et classification

Reference number
ISO 9689:1990(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 9689 was prepared by Technical Committee ISO/TC 172, *Optics and optical instruments*.

It 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).

Annex A of this International Standard is for information only.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Raw optical glass — Resistance to attack by aqueous alkaline phosphate-containing detergent solutions at 50 °C — Testing and classification

1 Scope

This International Standard specifies a method for testing the resistance of raw optical glasses to attack by aqueous alkaline phosphate-containing detergent solutions (phosphate solutions) at 50 °C and a classification of optical glasses according to the aqueous alkaline phosphate-containing detergent resistance (phosphate 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 3585:1976, *Glass plant, pipeline and fittings — Properties of borosilicate glass 3.3.*

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

3 Principle

Attack on polished glass by an aqueous solution containing 0,01 mol/l tripolyphosphate 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 the apparent attack to a depth of 0,1 µm with time scales given in a classification table to obtain the phosphate 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 Phosphate solution.

4.2.1 Purifying of commercially available tripolyphosphate

Prepare an aqueous solution containing 10 % (*m*/*m*) to 15 % (*m*/*m*) tripolyphosphate, filter out any insoluble matter and add ethanol (4.5) gradually, until one volume of ethanol has been added to four volumes of the phosphate solution. Stir for 30 min and filter off the hexahydrate crystals, wash twice with a mixture of one volume of ethanol and one volume of water and redissolve in a minimum amount of water. Repeat the process at least four times. Then dry the crystals at room temperature at a relative humidity of (50 ± 10) %. The yield is about 40 % to 45 % (based on the initial commercially available substance) and has impurities of less than 0,5 %.

4.2.2 Test solution

Dissolve 47,6 g of the purified tripolyphosphate (4.2.1) in water (4.1) in a one-mark volumetric flask having a capacity of 1000 ml; make up to volume and mix well. The solution contains approximately 0,1 mol/l phosphate. For the test, dilute one volume of the solution prepared with nine volumes of water (4.1). This new solution is called the test solution, the concentration of which is $c(Na_5P_3O_{10}) \approx 0{,}01$ mol/l; the pH shall be 10 ± 0,1 at 20 °C. If the pH value is different, the salt shall be purified again.

4.3 Nitric acid (HNO_3), analytical grade.

Dilute to pH = 4,5 ± 0,1.

4.4 Sodium hydroxide solution, $c(NaOH) \approx 0{,}1$ mol/l.

4.5 Ethanol (C_2H_5OH), volume fraction ϕ = 96 %, extra pure.

4.6 Isopropyl alcohol (C_3H_7OH).

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

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 (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 30 litres to 40 litres capacity, thermostatically controlled to maintain the temperature of (50,0 ± 0,2) °C.

5.5 Analytical balance, accurate to ± 0,1 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 diameters 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 Polishing

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 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 ± 3) °C. Each beaker shall contain sufficient isopropyl alcohol (4.6) 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 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 4 For drying, a drying oven may also be used, for 30 min at (115 ± 5) °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.

7 Procedure

7.1 General

The prepared samples shall only be used once.

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

Place the test vessel (5.1), filled with 2 litres of test solution (4.2.2) 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 ± 0,2) °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 as m_1, to an accuracy of ± 0,1 mg. Always use two samples of the same glass for the 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 rod 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. Take 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 isopropyl alcohol (4.6) 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 isopropyl alcohol (4.6) and dry by moving in air (see also note 4 in 6.3). Transfer the clean sample to 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 ± 0,1 mg. Calculate the time 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 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 °C.

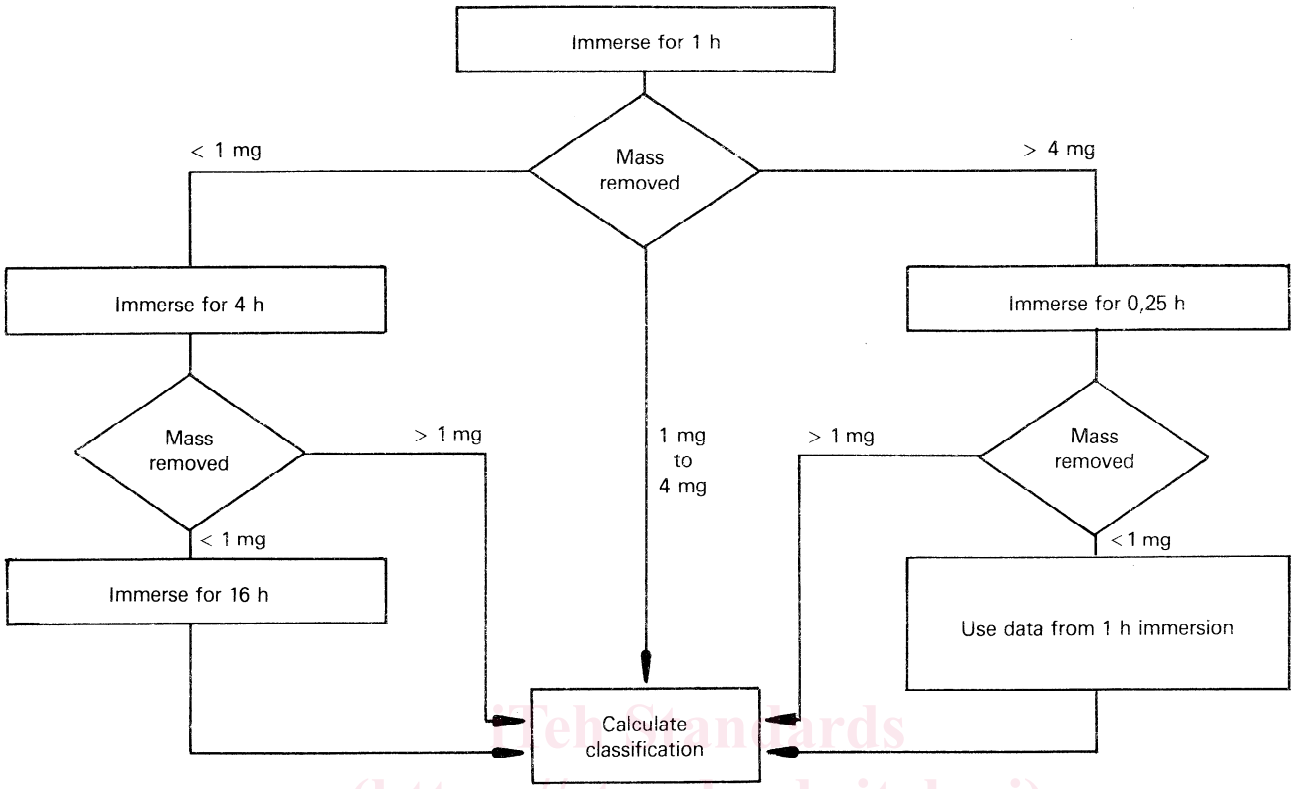

Figure 1 — Sequence of testing the phosphate resistance of an unknown optical glass

7.2 Testing unknown glasses

For this purpose 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 (4.2.2) 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 the 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 according to 7.3.

7.3 Testing known glasses

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

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 phosphate resistance class.