

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)
Documents
AVA Document Sharing Hub

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.

© ISO 1990

All rights reserved. No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

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 $c(\text{Na}_5\text{P}_3\text{O}_{10}) \approx 0,01 \text{ mol/l}$; the pH shall be $10 \pm 0,1$ 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 $\text{pH} = 4,5 \pm 0,1$.

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

4.5 Ethanol ($\text{C}_2\text{H}_5\text{OH}$), volume fraction $\phi = 96 \%$, extra pure.

4.6 Isopropyl alcohol ($\text{C}_3\text{H}_7\text{OH}$).

After evaporation of 100 ml of the alcohol, no residue shall be visible. If this is not the case, re-distill 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 $(50,0 \pm 0,2) \text{ }^\circ\text{C}$.

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 diameters to an accuracy of $\pm 1 \%$.

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 $\times$ 30 mm $\times$ 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 : $\approx 50 \%$
- 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).