### INTERNATIONAL STANDARD

ISO 8424

Third edition 2023-07

# Raw optical glass — Resistance to attack by aqueous acidic solutions — Test method and classification

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

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#### **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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see <a href="https://www.iso.org/directives">www.iso.org/directives</a>).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see <a href="https://www.iso.org/iso/foreword.html">www.iso.org/iso/foreword.html</a>.

This document was prepared by Technical Committee ISO/TC 172, *Optics and photonics*, Subcommittee SC 3, *Optical materials and components*.

This third edition cancels and replaces the second edition (ISO 8424:1996), which has been technically revised.

The main changes are as follows:

- a new measurement procedure, the powder method, was added;
- Annex A was added;
- Annex B was added;
- the surface method is technically revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

#### Introduction

This document specifies methods for testing the resistance of optical glasses to the attack by aqueous acidic solutions and provides corresponding classifications according to the resistance determined.

Two different methods are provided: A surface method and a powder method. Both methods are described side by side so that the user can select a suitable or convenient method for the application.

The surface method is applied to polished glass samples. The results are comparable to application conditions.

The powder method uses small amounts of crushed granular glass for testing. It is easy to apply, and provides test results quickly.

The acid resistance classes determined by the two different methods show a correlation, but they cannot be converted into each other unambiguously. Therefore, different notations are introduced for the acid resistance classes referring to the determining method to avoid misunderstandings.

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### Raw optical glass — Resistance to attack by aqueous acidic solutions — Test method and classification

#### 1 Scope

This document specifies two methods for testing the resistance of raw optical glasses to attack by aqueous acidic solutions and defines a classification of optical glasses according to the acid resistance determined by these methods.

The surface method tests the resistance of the polished plate-shaped optical glass to attack by aqueous acidic solutions at  $25\,^{\circ}\text{C}$  for a specified time and indicates the class determined by this method as "SR-S".

The powder method tests the resistance of crushed granular optical glass to attack by an acidic aqueous solution at above 98 °C for 1 h, and indicates the class determined by this method as "SR-P".

#### 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 3310-1, Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth

ISO 3585, Borosilicate glass 3.3 — Properties

ISO 4797, Laboratory glassware — Boiling flasks with conical ground joints

ISO 4799, Laboratory glassware — Condensers

ISO 10110-8, Optics and photonics — Preparation of drawings for optical elements and systems — Part 8: Surface texture

#### 3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <a href="https://www.iso.org/obp">https://www.iso.org/obp</a>
- IEC Electropedia: available at <a href="https://www.electropedia.org/">https://www.electropedia.org/</a>

#### 4 Principle of measurement

#### 4.1 Surface method

A polished glass sample is exposed to a test solution with a pH of 0,3 (nitric acid Solution 0,5 mol/l) or 4,6 (buffer solution) at 25 °C for specified durations. The sample is weighed before and after immersion to determine the loss in material. From this result the duration required to remove a surface layer of 0,1  $\mu$ m depth is determined and categorized into the acid resistance class SR-S.

#### 4.2 Powder method

The glass is ground into particles with diameters in the range of 425  $\mu m$  to 600  $\mu m$ . A powder sample equivalent to the specific gravity in grams is placed in a platinum basket. The basket is placed in a flask of silica glass and boiled for 60 min. The degree of the acid resistance is determined by measuring the mass loss (in percentage) and is categorized into the acid resistance class SR-P.

#### 4.3 Selection help for the methods

The pros and cons of both methods are listed in <u>Table 1</u>.

Annex A gives an informative overview on the comparability of the results of both methods.

**Pros** Cons Surface method The acid resistance class can be The test shall be repeated until the categorized "in detail" through several mass loss achieved is within the specified value. test steps. Appearance can be judged. Test duration cannot be predicted in advance for unknown glasses. It can be tested in the same condition as a polished product since the glass sample Tying is bothersome work when the is polished on all 6 faces. wire has been damaged. Closer to real application as in terms of surface tested. acid resistance Powder method class can be Appearance evaluation is not possible. categorized "simply". A specific Pt basket is required. The test duration is constant and short for all glasses. g/stand The test procedure (method) is simple.

Table 1 — Pros and cons of both methods

#### 5 Reagents

Use only reagents of recognized analytical grade.

#### 5.1 Nitric acid

- a) surface method: solution [ $c(HNO_3) = 0.5 \text{ mol/l}$ ], pH 0.3 + 0.05.
- b) powder method: solution [ $c(HNO_3) = 0.01 \text{ mol/l}$ ], pH 2,2 ± 0,05.
- **5.2** Acetic acid [CH<sub>3</sub>COOH =1,05 g/cm<sup>3</sup>], 100 % [mass fraction].
- **5.3 Sodium hydroxide**, solution [c(NaOH) = 1 mol/l].

#### 5.4 Alcohol

- a) surface method: 2-propanol (C<sub>3</sub>H<sub>7</sub>OH) is used. After evaporation of 100 ml of the alcohol, no residue shall be visible. If residue is still visible, re-distill 2-propanol;
- b) powder method: ethanol, methanol and 2-propanol can be used, but water-containing alcohol cannot be used.

#### **5.5 Buffer solution**, pH $4.6 \pm 0.05$ .

In glass vessels with graduated volumes (e.g. volumetric flask, beaker, conical beaker), mix 11,8 ml of the acetic acid (5.3), 200 ml of water and 100 ml of the sodium hydroxide solution (5.4). Fill up to the mark with water. Store in a plastic or borosilicate glass bottle.

#### 6 Surface method

#### 6.1 Apparatus

Usual laboratory equipment, together with the following.

**6.1.1 Beaker,** flat flange, made of borosilicate glass 3.3, in accordance 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 1).

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#### Key

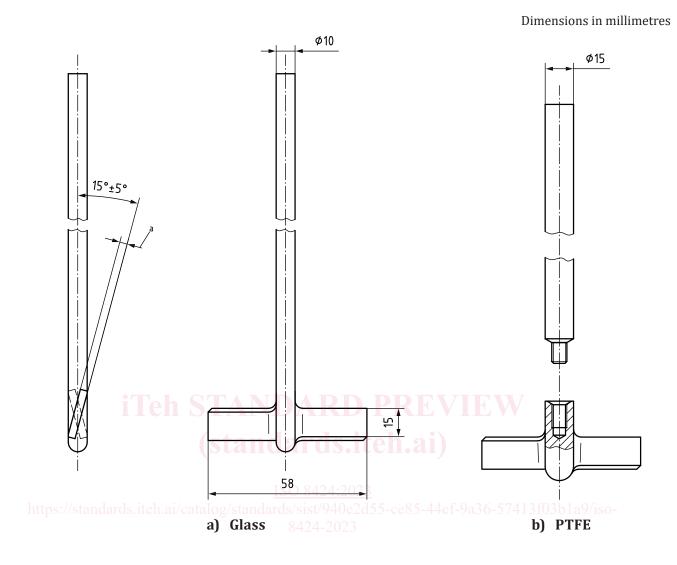
- 1 polyethylene foam
- 2 flat flange beaker (capacity of 2 000 ml)
- 3 stirrer (See Figure 3)
- 4 sample

- 5 sample
- 6 surface of the liquid
- 7 polymethyl methacrylate (PMMA) plate

Permissible variations in dimensions without tolerance indication shall be in accordance with the coarse series in ISO 2768-1.

 $Figure \ 1 - Test \ apparatus$ 

**6.1.2 Stirrer,** approximately 350 mm long, having a 10 mm diameter glass shaft, or a 15 mm diameter polytetrafluoroethylene (PTFE) shaft (see Figure 2) or the shaft made of similar material in workability, chemical stability and mechanical strength, e.g. polyether ether ketone (PEEK).



#### a 3 for glass stirrer, 5 for PTFE stirrer.

Permissible variations in dimensions without tolerance indication shall be in accordance with the coarse series in ISO 2768-1.

Figure 2 — Stirrers

#### **6.1.3** Acid resistant wires (e.g. platinum, nylon, etc.)

Less than 0,15 mm in diameter used to entwine the sample and hold it in the bath in such a way that the area that is not in contact with the test liquid is less than 1 %. It is also possible to use a different material as long as it is resistant to the test solution.

#### 6.1.4 Heating bath

Gas or electrically heated, with a capacity of 30 l to 40 l, thermostatically controlled to maintain the temperature of 25,0  $^{\circ}$ C ± 0,2  $^{\circ}$ C.

#### 6.1.5 Analytical balance

Accurate to  $\pm 0.1$  mg or better.