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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

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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 www.iso.org/directives).

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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 www.iso.org/iso/foreword.html.

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.

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 °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 https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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 µm 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 µm to 600 µm. 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 Table 1.

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

Table 1 — Pros and cons of both methods

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

5 Reagents

Use only reagents of recognized analytical grade.

5.1 Nitric acid

a) surface method: solution [$c(HNO_3)$ = 0,5 mol/l], pH 0,3 + 0,05.

b) powder method: solution [$c(HNO_3)$ = 0,01 mol/l], pH 2,2 ± 0,05.

5.2 Acetic acid [CH_3COOH =1,05 g/cm^3], 100 % [mass fraction].

5.3 Sodium hydroxide, solution [c(NaOH) = 1 mol/l].

5.4 Alcohol

a) surface method: 2-propanol (C_3H_7OH) 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 ± 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.

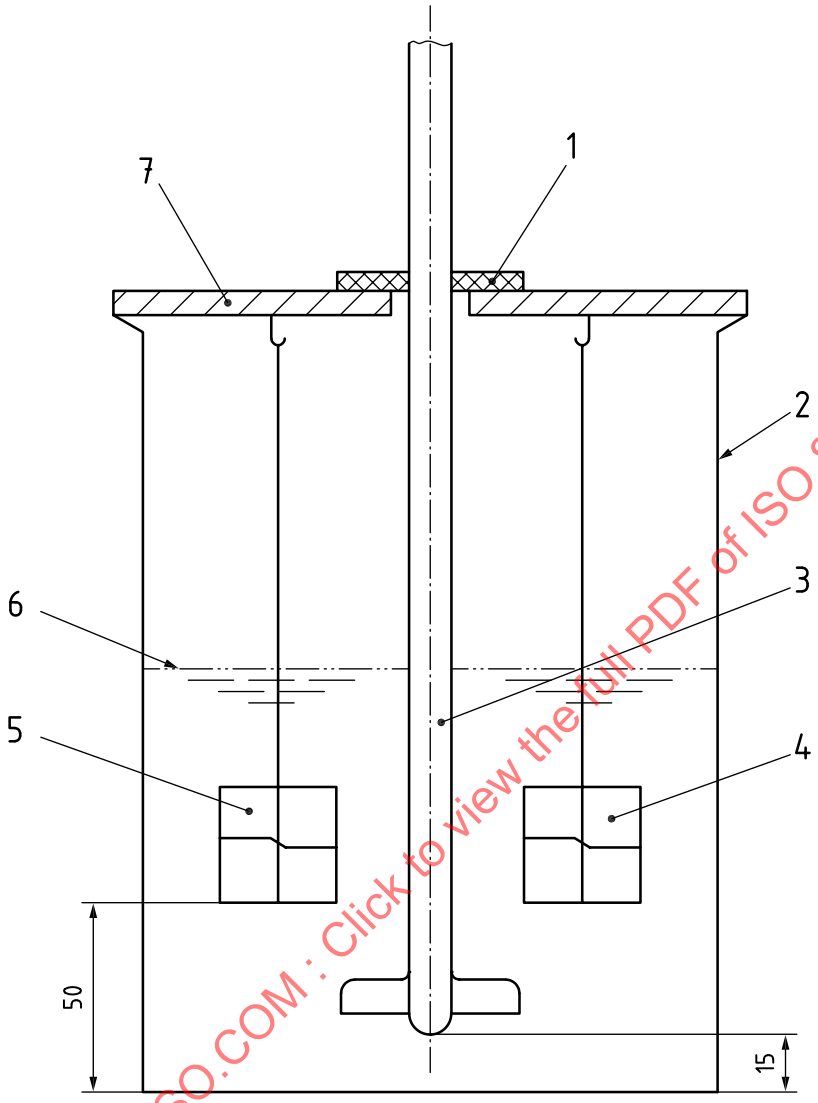
6 Surface method

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).

Dimensions in millimetres



Key

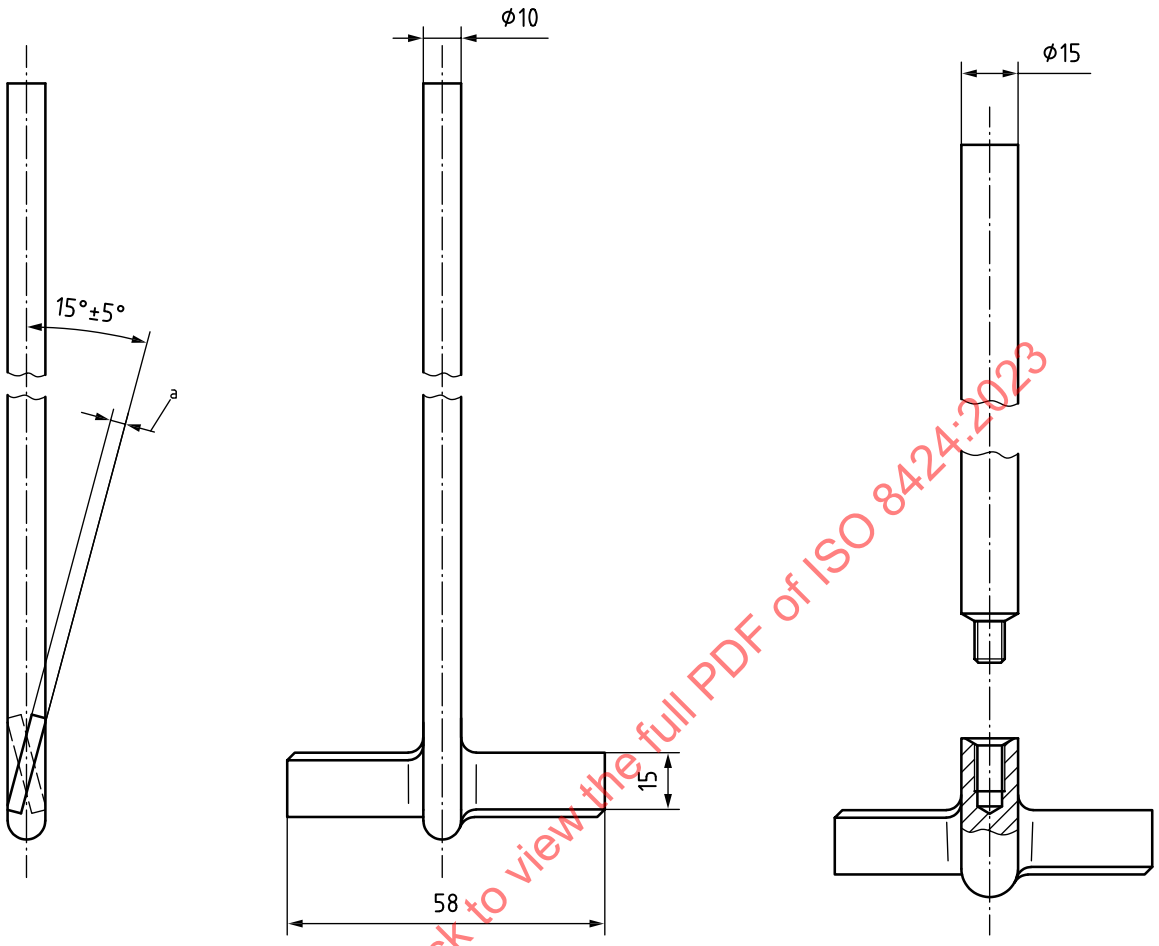
1 polyethylene foam
2 flat flange beaker (capacity of 2 000 ml)
3 stirrer (See Figure 3)
4 sample
5 sample
6 surface of the liquid
7 polymethyl methacrylate (PMMA) plate

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).

Dimensions in millimetres



a) Glass **b) PTFE**

[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 °C ± 0,2 °C.

6.1.5 Analytical balance

Accurate to ±0,1 mg or better.

6.1.6 Desiccator

Using a 2:1 mixture of silica gel (for H_2O absorption) and soda lime (a mixture of CaO and NA_2O, for CO_2 absorption) with indicator for regeneration.

6.1.7 Tongs

Protected with inert smooth material, for example plastics.

6.1.8 Measuring instruments

Suitable for measuring lengths and diameter to an accuracy of ±1 %.

6.1.9 Ultrasonic equipment for laboratory use

Filled with water, which can be heated to at least 50 °C.

6.1.10 Beakers, made of borosilicate glass 3.3 in accordance with the requirements of ISO 3585, having a capacity of 100 ml.

6.1.11 pH meter, it shall be calibrated with pH standard buffer before use.

6.2 Preparation of the samples

6.2.1 General

Cut pieces of the annealed glass (see ISO 9802) to be tested so that after polishing has been completed the dimensions are nominally (30 ± 1) mm × (30 ± 1) mm × (2,5 ± 0,5) mm. Apply the following polishing procedure to all surfaces of the samples using slurry made with water. The quality of the polished glass surfaces shall be in accordance with grade P1 requirements of ISO 10110-8. The target of the polishing process is to remove the sub-surface damage (micro cracks) of the preceding grinding process.

NOTE For polishing methods, see Annex B.

6.2.2 Lapping

The lapping should be achieved by using loose abrasive alumina or Silicon carbide, FEPA F600 or JIS R 6001-2 #1500.

6.2.3 Polishing

The polishing should be achieved by using cerium(IV) oxide abrasive having grains smaller than 2 µm and a suitable polisher. The polishing duration should be less than 30 min.

Flatten the sharp edges by slight polishing (chamfer).

Store the samples in the desiccator until they are needed for further processing.

Soda lime can erode the glass surface. Great care should be exercised in removing the desiccator lid so as not to disturb any dust.

6.2.4 Calculation of total surface area

Measure all dimensions with 0,2 mm or better accuracy and calculate the actual total surface area to an accuracy of 2 %.

Numerical rounding should be done after calculation of the surface area only.

Record the value obtained.

6.2.5 Cleaning

Samples shall be cleaned as soon as possible after polishing. For this purpose, place three 100 ml beakers in an ultrasonic water bath, containing water heated to 45 °C ± 3 °C. Each beaker shall contain sufficient 2-propanol 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 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 sequentially 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 can also be used for 30 min at 115 °C ± 5 °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.

6.3 Procedure

6.3.1 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 filled with 2 l of test solution in the heating bath, adjust the stirrer so that it is 15 mm above the vessel bottom and allow the temperature to reach 25,0 °C ± 0,2 °C. Transfer the cleaned samples, which have been cooled to room temperature in the desiccator, to the analytical balance using the tongs. Weigh and record the mass as m_1, to an accuracy of ±0,1 mg. Always use two samples of the same glass for one test in the same test beaker.

Entwine the platinum wire around the samples 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 1). There shall be no contact between the sample and the equipment.

Stir with a frequency of 100 r/min.

Duration of exposure shall be counted from the moment the samples are immersed in the test solution.

After the exposure duration, remove the samples from the liquid, wash twice with distilled water and remove the platinum wires. Immerse the samples three times into 2-propanol and dry by moving in air (see also NOTE in 6.2.5). 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 ±0,1 mg. Calculate the duration necessary for material removal depth of 0,000 01 cm (0,1 µm) in accordance with the formula given in 6.4 and observe the changes in the glass surface (see 6.4 and 6.5).

NOTE Observation of the glass surface is performed under natural light or under illumination by a microscope lamp at an angle of approximately 45°.

6.3.2 Testing unknown glasses

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

Prepare six samples in accordance with 6.2 and follow the test sequence shown in Figure 3 for each sample. The test should be performed on one at a time.

Start the test by immersing one sample into the test solution for 16 h. Depending on the loss in mass, calculate the classification or continue with the next exposure with the same pH for 100 min or pH 0,3 for 16 h (see sequence in Figure 3).

The class is typically calculated with 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 exposure.

When the conditions for exposure have been determined, continue according to 6.3.3 (see Table 2).

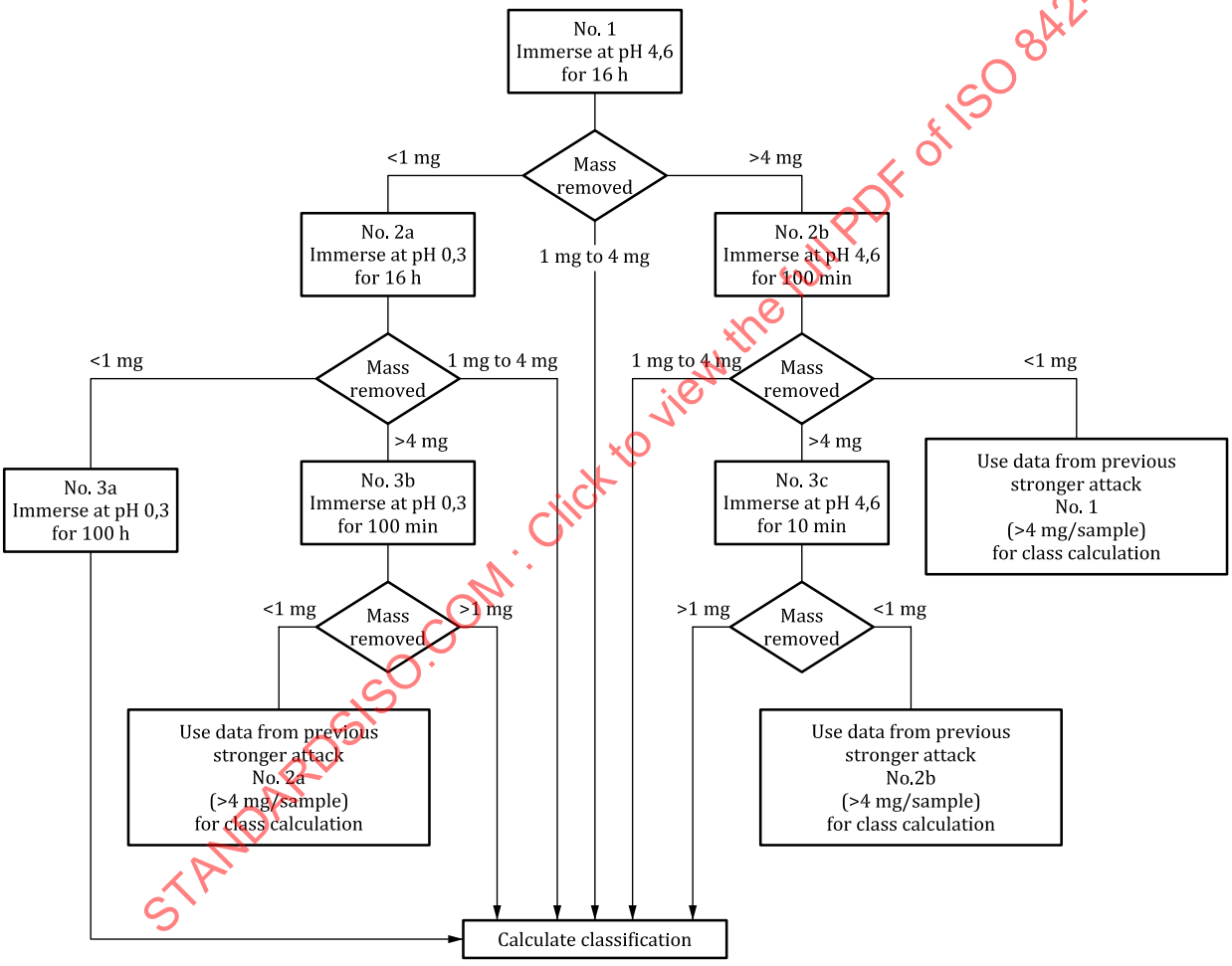


Figure 3 — Sequence of testing the acid resistance of an unknown optical glass

6.3.3 Testing known glasses

If the acid resistance class of an optical glass is reasonably well-known or determined in accordance with 6.3.2, the procedure given in 6.3.1 under the conditions specified in Table 2 shall be applied.

The results from exposure 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 less than 1 mg/sample, proceed with the next stronger attack. If the loss in mass is more than 4 mg/sample, proceed with the next weaker attack (see Figure 3).

Table 2 — Different solutions and specified exposure duration for attack of optical glasses in the acid resistance test with approximate acid resistance class SR-S

Test solution No.	Test solution pH	Exposure duration min (h)	Approximate acid resistance class SR-S[a]	
3c	4,6	10 (0,17)	Weakest attack	53
2b	4,6	100 (1,67)	Weaker	52
1	4,6	(16)	Weak	51 or 5
3b	0,3	100 (1,67)	Strong	4 or 5
2a	0,3	(16)	Stronger	3
3a	0,3	(100)	Strongest attack	2 or 1

[a] The correct acid resistance class SR-S according to Table 4 is calculated by Formula (1) given in 6.4 using the loss in mass determined according to 6.3.3.

6.4 Expression of results

From the mean value of the loss in mass determined in accordance with 6.3, calculate the duration, t_d in hours, necessary to etch a surface layer to a depth of 0,000 01 cm (0,1 µm) using the following Formula (1):

$$t_d = \frac{t_e \times \rho \times A \times d}{m_1 - m_2} \quad (1)$$

where

t_d is the time needed to etch to a depth of 0,000 01 cm (= 0,1 µm);

d is the surface layer etch depth in cm, 0,000 01 cm (= 0,1 µm);

t_e is the duration of exposure to the acid in the experiment, in hours;

ρ is the density of the glass, in grams per cubic centimetres;

A is the total surface area of the sample, in square centimetres;

m_1 is the mass of the sample before the acid attack, in grams;

m_2 is the mass of the sample after the acid attack, in grams.

6.5 Classification and designation

Optical glasses shall be classified in accordance with Table 3, according to the duration, t_d in hours, necessary to etch a surface layer to a depth of 0,1 µm when tested by the method specified in this document.

Table 3 — Classification of optical glasses

Acid resistance class SR-S (Class#)	pH of the acid solution	Exposure duration needed to etch to a depth of 0,1 µm (t_d in 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 document, the designation shown in the following example shall be used.

Changes in the surface of the sample that are visible after determining the mass m_2 (see 6.3.1) used for the calculation (see 6.4) are qualitatively evaluated with the naked eye and added to the class number (Class#) as follows:

Class#.0: no visible changes;

Class#.1: clear, but irregular marked, pitted (surface wavy, pock-marked, pitted);

Class#.2: staining and/or interference colours (slight selective leaching):

Class#.3: tenacious thin whitish layer (stronger selective leaching, a cloudy/hazy/dullish surface);

Class#.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 6.2.2 and 6.2.3) may be responsible for a deviation of one place in the additional numbers to the class.

EXAMPLE For a glass having a density ρ = 3,31 g/cm^3, a total surface area A = 20,4 cm^2, a loss in mass $m_1 - m_2$ = 3,7 mg/sample, after an exposure duration t_e = 100 min (= 1,67 h) by an acid solution of pH 0,3, resulting in to t_d = 0,30 h for a material removal to a depth of 0,1 µm and with interference colours visible after the attack:

Optical glass, acid resistance class ISO 8424 SR-S 4.2

7 Powder method

7.1 Apparatus

Usual laboratory equipment, together with the following.

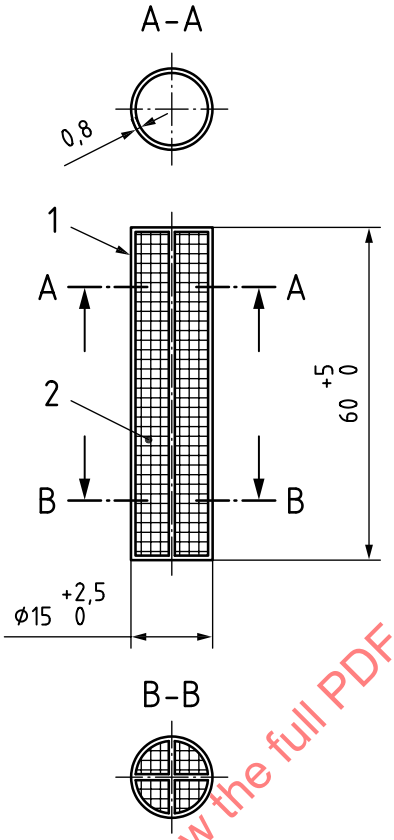
7.1.1 Sieve

The sieve shall refer to the test sieve specified in ISO 3310-1.

7.1.2 Basket for corrosion test

The mesh basket for the corrosion test shall be fabricated with a specific opening size between 230 µm and 260 µm using platinum wire with a diameter about 76 µm. A drawing of the apparatus including dimensions is shown in Figure 4.

Dimensions in millimetres



Key

1	platinum frame
2	platinum test sieve
A-A	cross-section view
B-B	cross-section view

Figure 4 — Basket

7.1.3 Apparatus for corrosion test

The configuration and dimensions of the apparatus are shown in Figure 5. However, the dimensions of Figure 5 are merely an example, the shape shall conform to ISO 4797 and ISO 4799. The dimension and shape may be different, as long as the nominal capacity of a spherical-shaped flask is 100 ml. The apparatus shall employ a flask fitted to a condenser made of either fused silica glass or fused borosilicate glass as specified in ISO 4797 and ISO 4799.

Dimensions in millimetres

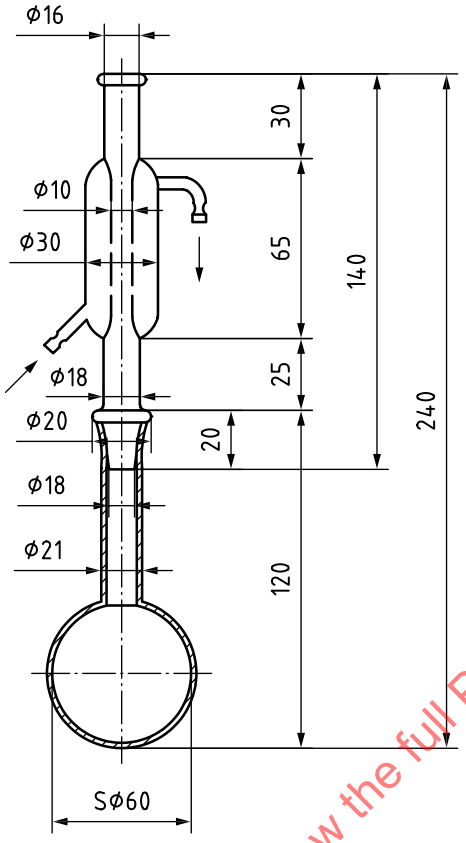


Figure 5 — Apparatus for corrosion

7.1.4 Heating bath

The apparatus shall use a water bath in which the spherical part of the flask is completely immersed in hot water and shall be held at a temperature above 98 °C measured at 20 mm ± 10 mm from the bottom of the flask.

7.2 Preparation of the specimen of the glass to be tested

Prepare the specimen as follows:

a) Crush the glass and pass through a screen with a 710 µm sieve and pass the selected particles again with a 600 µm sieve. Then reselect the glass particles with a 425 µm sieve to collect particles of sizes from 425 µm to 600 µm.

b) Place the powdered glass weighing approximately three times its specific gravity in grams into a 50 ml beaker. To remove dust powder from the particles, pour 15 ml of alcohol for decantation.

c) After repeating operation b) five times, dry the specimen at a temperature between 120 °C and 130 °C in an oven for one hour and then store it in a desiccator with silica gel.

7.3 Procedure

Use the following procedure:

a) Place a specimen weighing as much as its specific gravity in grams in the basket. Shake the basket gently and then place it in a weighing bottle with a lid and carefully weigh it.

b) Pour 80 ml of 0,01 mol/l "0,01 N" nitric acid into the clean and dried flask coupled to a condenser. Then place the flask in the heating apparatus for 10 min.

c) Gently place the basket containing the specimen in the flask. After heating for 60 minutes in the apparatus, remove the basket.

d) Pour 80 ml of alcohol into a 100 ml beaker. Then soak the basket in the alcohol and rinse it.

e) After repeating operation d) three times, the basket shall be put in a weighing bottle and dried in an oven at the temperature between 120 °C and 130 °C for 1 h.

f) Cool the weighing bottle for one hour in a desiccator with silica gel and weigh it carefully together with a lid.

The procedures described above, from a) to f), shall be repeated twice.

The percentage mass loss (%) is obtained from the initial mass of the specimen and the total mass. Two individual measurements are carried out to obtain the mean value.

7.4 Classification and designation

Optical glasses shall be classified by their mass loss in accordance with Table 4.

Table 4 — Acid resistance classification of optical glasses based on the powder method (SR-P)

Acid resistance (SR-P)	1	2	3	4	5	6
Mass loss %	<0,20	<0,35	<0,65	<1,20	<2,20	≥2,20

EXAMPLE For a glass with an initial mass of 4,360 90 g and after applying the powder test method a mass loss in the first test of 0,071 17 g and 0,068 87 g in the second test, the resulting average percentage mass loss is 0,68 % glass (See Table 5 with example values). This glass is then categorized into the acid resistance class SR-P 4 and its designation is as followed:

Optical glass, acid resistance class ISO 8424 SR-P 4

Table 5 — Example of powder test report entry

Glass Name	First test	Second test
Specific gravity	5,89	
Basket mass (g)	5,888 32	5,904 63
Initial mass of specimen (g)	4,360 90	4,360 83
Total mass before test (g)	10,249 24	10,265 46
Total mass after test (g)	10,178 07	10,196 57
Mass loss (g)	0,071 17	0,068 87
Percentage mass loss (%)	0,694 39	0,671 18
Average value of percentage mass loss (%)	0,682 79	
Acid resistance class SR-P	4	

8 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 8424:2023;

b) identification of the samples, including density;