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Optics and photonics — Optical materials and components — Test method for climate resistance of optical glass

Optique et photonique — Matériaux et composants optiques — Méthode d'essai pour la résistance climatique du verre optique

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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.

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

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.

Optics and photonics — Optical materials and components — Test method for climate resistance of optical glass

1 Scope

This document specifies the test method for climate resistance of optical glass and the classification of the optical glass according to the test results.

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 3585, *Borosilicate glass 3.3 — Properties*

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

ISO 14782, *Plastics — Determination of haze for transparent materials*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

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

3.1
haze
percentage of transmitted light, passing through a specimen, which deviates from the incident light by no more than 0,044 rad (2,5°) by forward scattering

[SOURCE: ISO 14782:1999, 3.1]

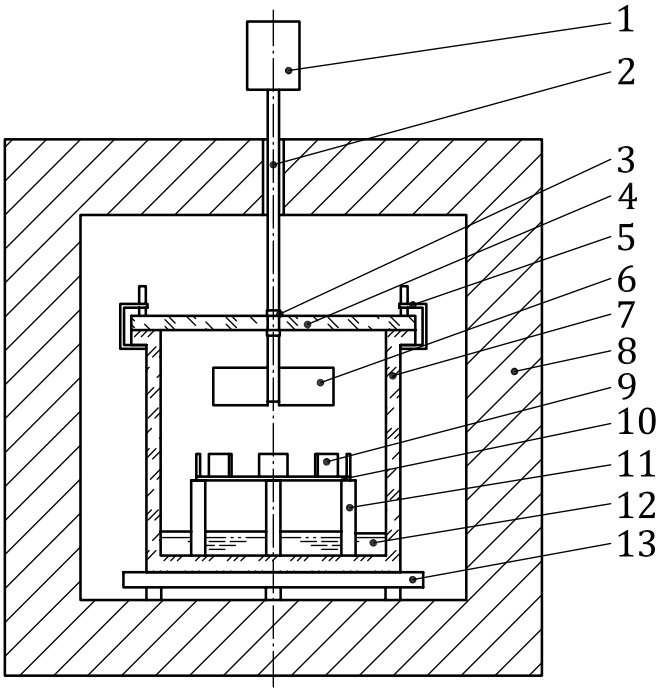
4 Principle

To evaluate the climate resistance of optical glass in its operating environment, the hazes of polished glass surfaces before and after testing are measured with the haze meter specified in ISO 14782, and the climate resistance is determined by the change in the amount of haze.

5 Test apparatus

5.1 Configuration

The test apparatus consists of the components shown in Figure 1. The size and arrangement of the components in the glass water tank are shown in Figure 2.

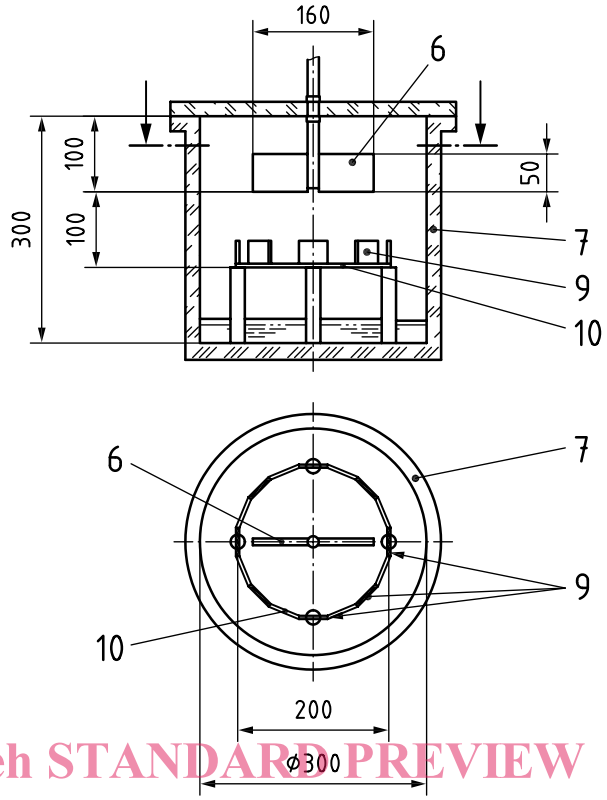


Key

1	stirring motor	8	thermostatic chamber
2	stirring rod	9	specimens
3	seal	10	specimen holder
4	lid	11	specimen holder stand
5	clamp	12	distilled water (1,8 l)
6	stirring fan	13	water tank stand
7	glass water tank		

Figure 1 — Test apparatus

Dimensions in millimetres



Key

6 stirring fan
7 glass water tank
9 specimens
10 specimen holder (stainless steel)

Figure 2 — Arrangement of specimens and units in the glass water tank

5.2 Thermostatic chamber

The chamber shall have an automatic temperature adjustment capability.

The ceiling of chamber shall have a through-hole for the stirring rod which is rotated by the stirring motor as shown in Figure 1.

5.3 Glass water tank and lid

The water tank and the lid shall be made of either borosilicate glass 3.3 in accordance with ISO 3585 or quartz glass, the thickness of which shall be between 5 mm and 20 mm. They shall be placed in the thermostatic chamber as shown in Figure 1.

Eliminate the gap of the contacting part between the lid and the rim of the tank by lapping. A hole shall be provided at the centre of the lid so that the stirring rod passes through it.

5.4 Water

The purity of the water used shall be in accordance with the grade 2 of ISO 3696. The amount of water contained by the water tank shall be 1,8 l.

5.5 Stirring unit

The stirring unit consists of a stirring motor, a stirring rod and a stirring fan as shown in Figure 1. The stirring rod shall be straight, not bent or warped. The dimension of the stirring fan is shown in Figure 2.

NOTE If the stirring rod is bent or warped, the stirring rod will be decentered during rotation and the stirring fan will be shifted from the correct position.

5.6 Seal

An elastic seal, such as an O-ring, shall be provided at the gap between the stirring rod and the lid to keep humidity in the tank constant during the test. Confirm the condition of the elastic seal before the test and exchange the seal if it deteriorates.

5.7 Specimen holder

Figure 3 shows an example configuration of a hexadecagonal specimen holder at the position of 100 mm under the stirring fan above the water level, which keeps the specimen's surface vertical. This holder shall be fabricated in a shape that allows up to eight specimens to be placed at equal intervals from the central axis of the stirring rod as shown in Figure 1.

The specimen holder shall be placed in the centre of the water tank, as shown in Figure 2.

Dimensions in millimetres

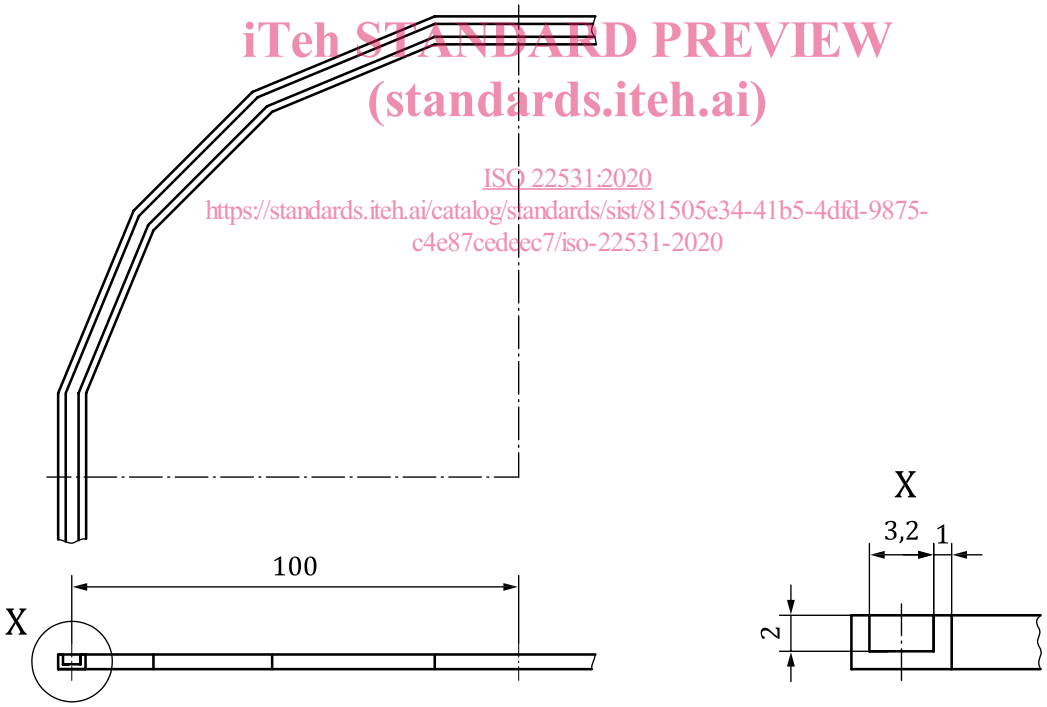


Figure 3 — Configuration example of the specimen holder

5.8 Specimen holder stand

The specimen holder stand shall hold the specimens 100 mm from the inner bottom of the water tank.

5.9 Water tank stand

Place a stand under the water tank to ensure a pathway of airflow.

6 Specimens

6.1 Shape and size of specimens

The shape of the specimen shall be a square-formed plate, and its size shall be 30 mm × 30 mm × 3 mm.

6.2 Number of specimens

The number of specimens per test shall be 5 to 8.

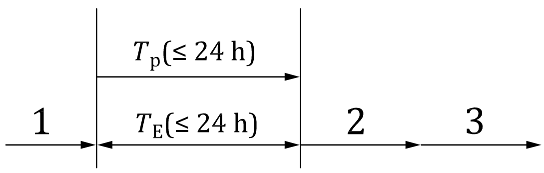
6.3 Surface treatment of specimens before test

Both sides of surfaces of each specimen shall be polished with cerium oxide slurry, then contamination shall be removed from the polished surfaces with an organic solvent. After that, the polished specimens are cleaned with detergent, distilled water and alcohols such as IPA (isopropyl alcohol). The detailed cleaning method shall be described in Annex B. The cleaning shall be started within 24 h after the polished surface has been exposed to the atmosphere, and after that, the test shall be started immediately.

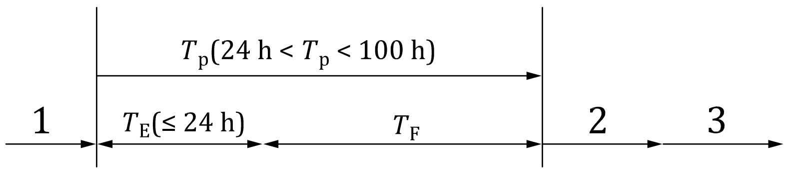
If the cleaning procedure of the specimens is started more than 24 h after polishing, the protective materials, i.e. the films or resins for the glass lens polishing process shall be applied to the polished surfaces immediately after polishing in order to avoid exposing the glass surface to the atmosphere. Dissolve the protective film with organic solvent or remove it before the cleaning of the specimens.

Even with the presence of protective materials, keeping moisture away from the glass perfectly is difficult, so the storage period of the specimens should not exceed 100 h. Further, if the glass surface changes with a protective material, cleaning and testing process shall be started promptly after polishing without using the protective material.

NOTE The duration to prepare for cleaning, i.e. T_P, changes depending on the time period from polishing to cleaning procedure, as shown in Figure 4. However, the duration that the specimens are exposed to the atmosphere, T_E, is within 24 h in each condition.



a) Condition 1: T_P within 24 h



b) Condition 2: T_P more than 24 h

Key

T_P the duration to prepare for cleaning
T_E the duration that the specimens are exposed to the atmosphere
T_F the duration that the protective films on the specimens are applied
1 polishing
2 cleaning
3 testing

Figure 4 — Schematic figure of time durations regarding the exposure time T_E of the specimen

7 Test method

7.1 Procedure of the test

The test shall be conducted in the following sequence.

a) Measure the haze of the pre-testing specimens using the haze meter specified in ISO 14782. Haze shall be measured more than 5 mm inside from the edge of the specimen.

b) With the stirring fan rotating at 100 r/min, set the temperature T_1 of the thermostatic chamber and keep for t_H, holding time, minutes or longer in order to achieve and maintain the air temperature within the water tank at 57,5 °C. The temperature T_1 and the holding time t_H, are determined in Annex A. Temperature tolerance should be ±0,5 °C.

c) Place the specimens into the holder so that the distance between specimens is at least 30 mm.

d) Put the lid on the tank and clamp them together.

e) Rotate the stirring fan at 100 r/min during the test.

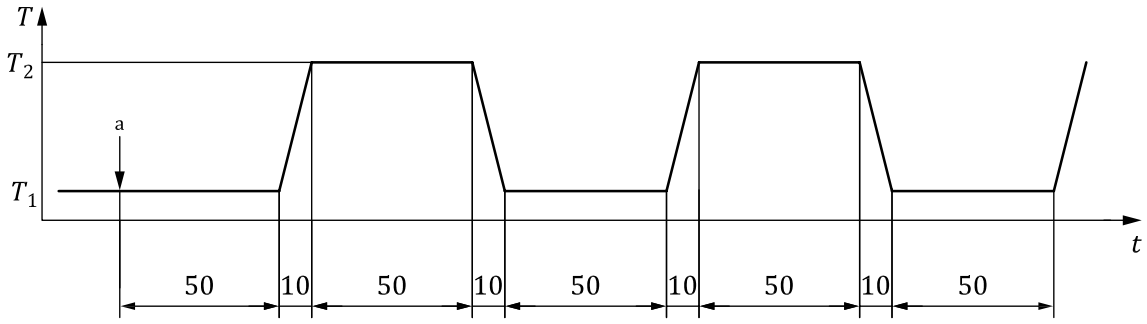
f) Maintain the air temperature in the water tank at 57,5 °C for 50 min.

g) Apply the temperature profile of the thermostatic chamber as shown in Figure 5 so that the actual air temperature in the water tank has a cycle in which the minimum temperature is 57,5 °C and the maximum temperature is 64,0 °C, as shown in Figure 6. To achieve such a temperature profile, use the set temperatures T_1 and T_2 determined in Annex A.

NOTE As shown in Figure 6, the actual air temperature change in the water tank is more moderate than the temperature cycle shown in Figure 5.

h) Apply the temperature cycle for 48 h, then remove the specimens and transfer them to a desiccator. Cool the specimens to room temperature and dry them.

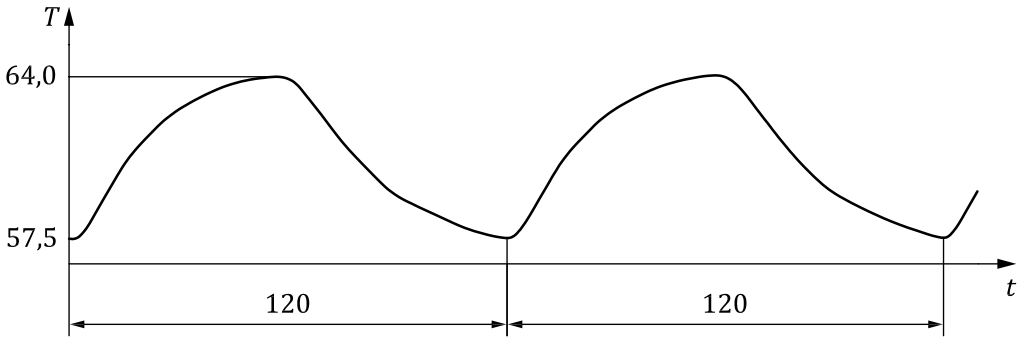
i) Measure the haze of the post-testing specimens using the haze meter. Haze shall be measured more than 5 mm inside from the edge of the specimen.



Key

T temperature (°C)
T_1 lower set temperature
T_2 upper set temperature
t time (min)
a Specimen set time.

Figure 5 — Temperature profile of thermostatic chamber



Key

t time (min)

T temperature (°C)

Figure 6 — An example of the measured air temperature profile in the water tank

7.2 Calculation

The change in the amount of haze shall be calculated from the following formula.

The change in the amount of haze for specimen No. k is $\Delta H(k)$ (k = 1, 2, 3..., n). This is shown in the following formula:

$$\Delta H(k)=H_{\mathrm{A}}(k)-H_{\mathrm{B}}(k) \tag{1}$$

where

$\Delta H(k)$ is the change in amount of haze for the k^{th} specimen (%);

$H_{\mathrm{A}}(k)$ is the haze of the k^{th} specimen after the test (%);

$H_{\mathrm{B}}(k)$ is the haze of the k^{th} specimen before the test (%).

Exclude the maximum value $\Delta H_{\max}$ and the minimum value $\Delta H_{\min}$ from the data to calculate the change amount of haze of each specimen $\Delta H(k)$, which is calculated from the haze measurements before and after the test. Calculate the average ΔH_{ave} from the remaining data, as shown in the following formula:

$$\Delta H_{\mathrm{ave}}=\left(\sum_{k=1}^{n}\Delta H(k)-\Delta H_{\max}-\Delta H_{\min}\right)/(n-2) \tag{2}$$

where

ΔH_{ave} is the average of change amount of haze $\Delta H(k)$ excluding the maximal value $\Delta H_{\max}$ and the minimal value $\Delta H_{\min}$ (%);

$\Delta H(k)$ is the change amount of haze for k^{th} specimen (%);

$\Delta H_{\max}$ is the maximal value of $\Delta H(k)$ in specimens from 1st to k^{th} (%);

$\Delta H_{\min}$ is the minimal value of $\Delta H(k)$ in specimen s from 1st to k^{th} (%);

n is the number of specimens.