INTERNATIONAL STANDARD

ISO 22531

First edition
2020-05

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

Reference number
ISO 22531:2020(E)

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Published in Switzerland

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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 www.iso.org/directives).

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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]

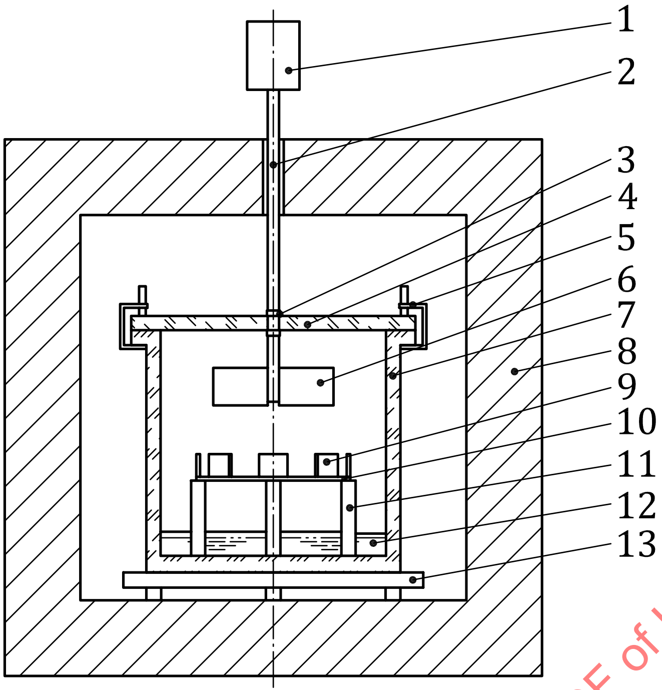
4 Principle

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.

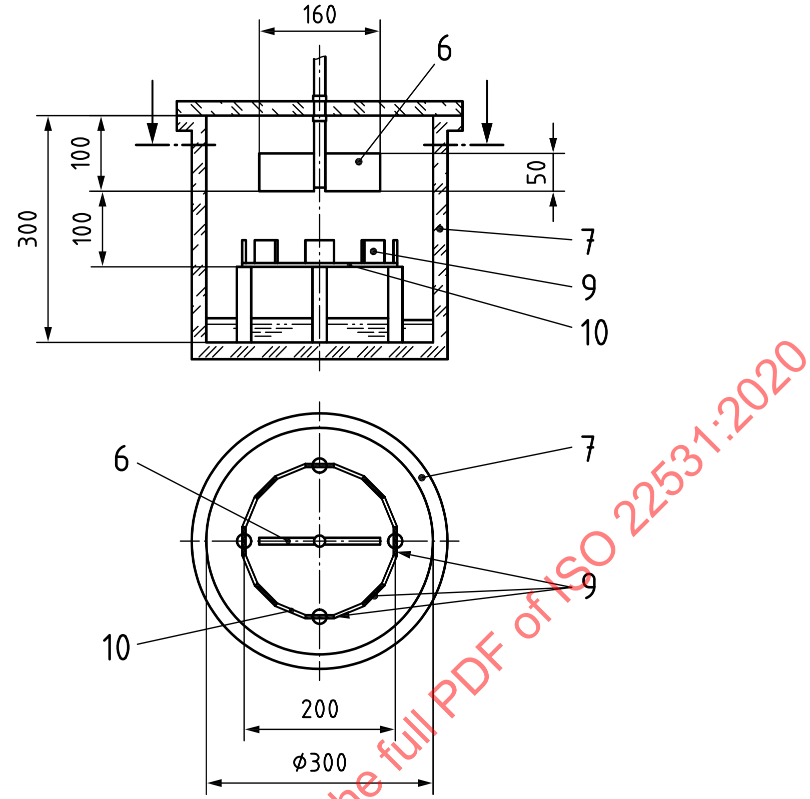


Key

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

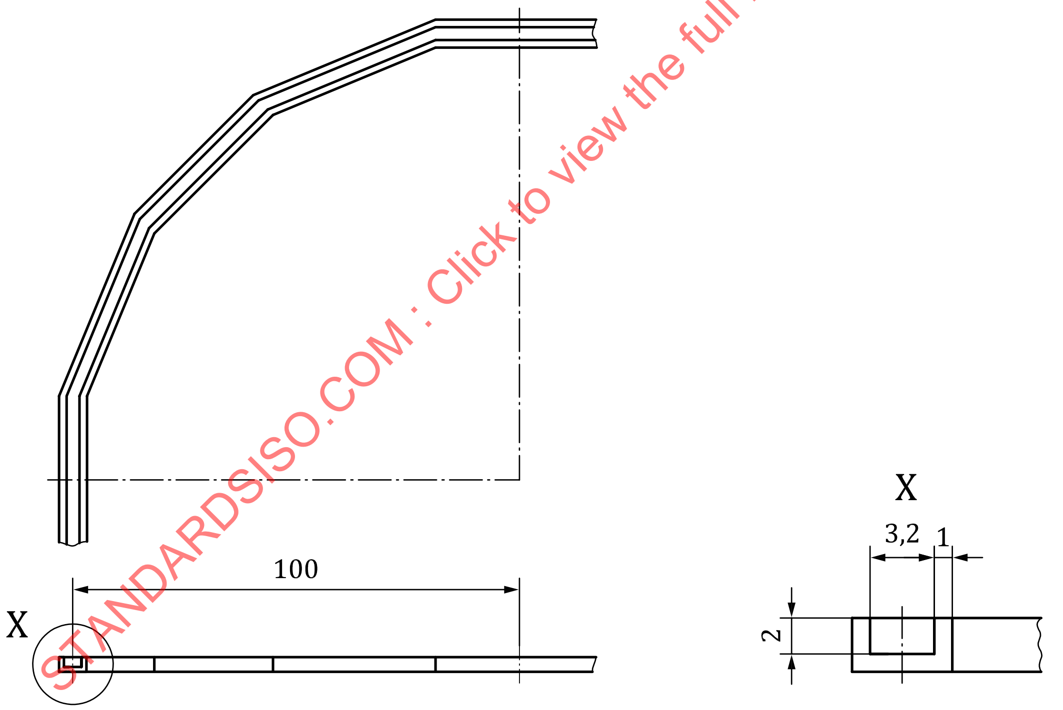


Figure 3 — Configuration example of the specimen holder

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.

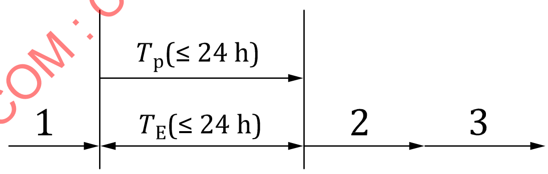
6.3 Surface treatment of specimens before test

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.

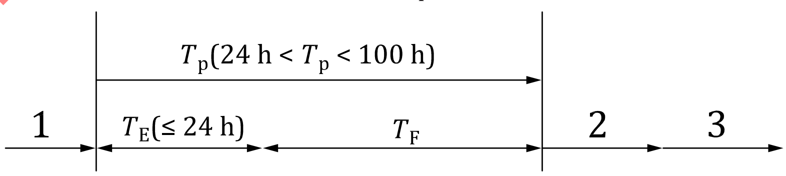
If the cleaning procedure of the specimens is started more than 24 h after polishing, the protective materials, i.e. the films or resins for the glass lens polishing process shall be applied to the polished surfaces immediately after polishing in order to avoid exposing the glass surface to the atmosphere.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

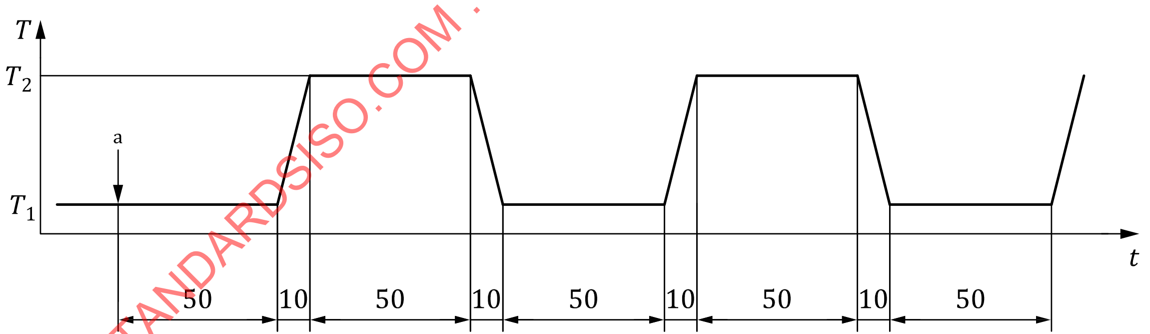
f) Maintain the air temperature in the water tank at 57,5 °C for 50 min.

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)

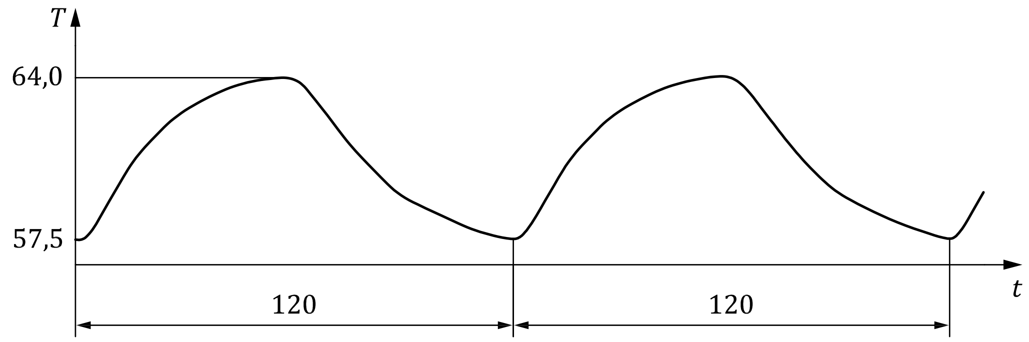
T_1 lower set temperature

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:

$$\Delta H(k) = H_A(k) - H_B(k) \quad (1)$$

where

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

$H_A(k)$ is the haze of the k^{th} specimen after the test (%);

$H_B(k)$ is the haze of the k^{th} specimen before the test (%).

Exclude the maximum value ΔH_{max} and the minimum value ΔH_{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:

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

where

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

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

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

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

n is the number of specimens.

EXAMPLE Table 1 shows an example of haze measurement results. According to Formula (2), exclude $\Delta H(3)$, which is the maximum value of $\Delta H(k)$, and $\Delta H(1)$, which is the minimum value, and average the remaining six values of $\Delta H(k)$. The resulting ΔH_{ave} is 47,4 %, as shown below:

$$\Delta H_{ave} = [\{\Delta H(1) + \Delta H(2) + \Delta H(3) + \Delta H(4) + \Delta H(5) + \Delta H(6) + \Delta H(7) + \Delta H(8)\} - \Delta H(1) - \Delta H(3)]/(8 - 2)$$

$$= (43,2 + 52,4 + 52,7 + 49,5 + 45,1 + 41,4)/6$$

$$= 47,4(\%)$$

Table 1 — Example of haze measurement results

Specimen No. k	Haze before test $H_B(k)$ %	Haze after test $H_A(k)$ %	Change in the amount of haze $\Delta H_A(k)$ %	Remarks
1	0,1	40,0	39,9	Minimum
2	0,1	43,3	43,2	
3	0,1	52,9	52,8	Maximum
4	0,1	52,5	52,4	
5	0,1	52,8	52,7	
6	0,1	49,6	49,5	
7	0,1	45,2	45,1	
8	0,1	41,5	41,4	

8 Classification and designation

Optical glasses shall be classified by the degree of climate resistance in accordance with Table 2.

Table 2 — Climate resistance classification of optical glasses

Class	ΔH_{ave} %
CR0	<0,3
CR1	<3
CR2	<10
CR3	<30
CR4	≥30

EXAMPLE According to Table 2, the climate resistance of the glass in the example in 7.2 is indicated as "Class 4", since the ΔH_{ave} of the glass is 47,4 %.

9 Test report

The test report shall include the following information:

a) the date of the test;

b) a reference to this document (including its year of publication), e.g. ISO 22531:2020;

c) an identification of the samples;

d) the method used (e.g. according to ISO 22531:2020);

e) a designation of the climate resistance;

f) any deviations from the procedure;

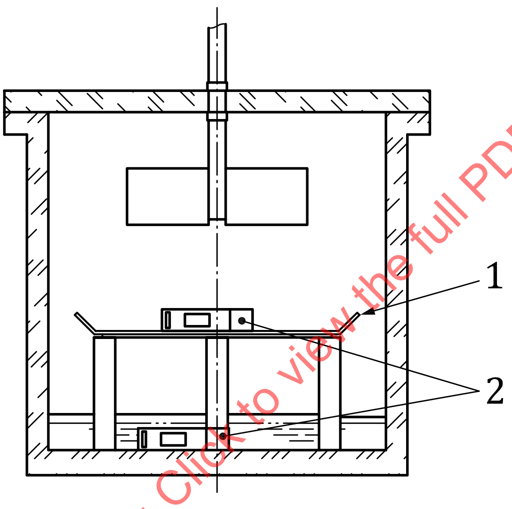
g) any unusual features observed.

Annex A
(normative)

Test conditions for the thermostatic chamber

Before starting the test, set up the thermostatic chamber according to the following procedures:

a) Place an air thermometer, e.g. stand-alone temperature data logger, at the holder position in the water tank, and place a water thermometer, e.g. stand-alone and immersive (waterproof) temperature data logger, in the water. The location of the thermometers in the glass water tank are shown in Figure A.1.



Key

1 tray

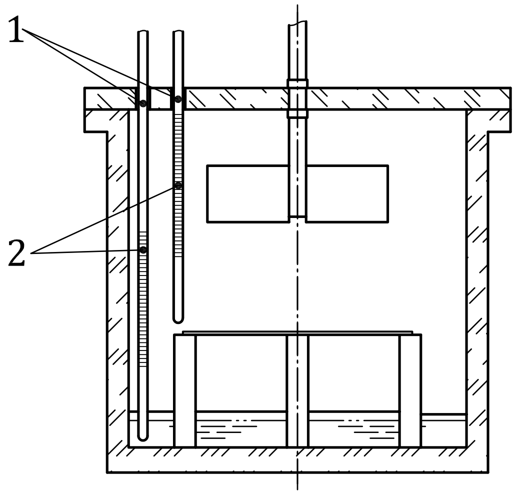
2 stand-alone temperature data logger

Figure A.1 — Example of the location of standalone and immersive thermometers

When the stand-alone thermometer is not available, two closable apertures could be constructed on the lid to allow the insertion of wired temperature sensors which allow air temperature and water temperature to be measured in the water tank. The insertion aperture should be positioned at a radial distance of 90 mm to 120 mm from the centre of the lid. In this case, the gaps of the apertures shall be closed during measurement so as not to lower humidity in the tank. The example of location of the thermometers are shown in Figure A.2.

The internal size of the chamber used in the round robin test was approximately 100 dm^3 to 200 dm^3 (100 l to 200 l). IEC/JIS 60068-3-5[2] can be referred as a general precaution when installing it. However, the specifications of the thermostatic chamber are not required in this document. Because if the temperature profile shown in Figure 5 can be achieved, the detailed provisions of the device itself are not required.

According to proposers' round robin tests, the temperature profile was able to reproduce if the wall of the water tank ranged from 5 mm to 20 mm. For example, the thickness can be chosen about 10 mm ± 2,5 mm considering handling and availability (not normative reference).



Key

1 closable apertures

2 wired temperature sensors

Figure A.2 — Example of the location of wired thermometer

b) Set the temperature in the thermostatic chamber to 57,5 °C and maintain it until both the water and air temperatures measured by two thermometers in the tank are the same at 57,5 °C. The time required to reach the target temperature is defined as a holding time t_H, described in 7.1 b).

c) Adjust the set temperatures T_1 and T_2 as shown in Figure 5 so that the air thermometer in the water tank indicates a minimum temperature of 57,5 °C and a maximum temperature of 64,0 °C. The method for adjusting temperatures is as follows:

  1) First, set T_1 to 57,5 °C and T_2 to 64,0 °C. Continuously implement the temperature cycle as shown in Figure 5.

  2) Measure the air temperature in the water tank during the temperature cycle with the thermometer. As the result of measurement, if the maximum and the minimum temperatures in the tank do not reach 64,0 °C and 57,5 °C respectively, adjust T_2 and T_1 so that these temperatures reach 64,0 °C and 57,5 °C.

If the measured maximum and minimum air temperatures in the water tank are 63,5 °C and 57,5 °C, respectively, T_2 shall be increased by 0,5 °C in order to reach the high temperature of 64,0 °C. Provided, however, that the lowest temperature in the water tank may increase to higher than 57,5 °C at the same time. Adjustment is required in such a case; for example, T_2 shall be increased by 0,5 to 1,0 °C, and T_1 shall be simultaneously decreased by 0,0 °C to 0,5 °C. This range of temperature adjustment is influenced by the capacity and heating ability of the thermostatic chamber, the thickness of the water tank, etc. Therefore, when changing the test apparatus and the testing environment, adjustments as described above are required in advance.

NOTE The temperatures of 57,5 °C and 64,0 °C and the holding time at each temperature were determined as the most effective conditions, i.e. the conditions with small errors and easy handling, based on the results of proposers' round robin tests. See also Reference [3].

Annex B
(normative)

Procedure for polishing and cleaning the specimens

B.1 Specimen polishing

The polished specimen is prepared as follows:

a) Polish both 30 mm × 30 mm faces with cerium oxide to a level such that no grinding streak can be observed by visual inspection under a 32 W cool white fluorescent lamp.

b) Immediately remove contamination from the polished surfaces with an organic solvent, etc.

c) Do not touch the polished surface of the specimen; instead, use a vertical holding jig to transfer the specimen in order to avoid contamination of the specimen surface.

d) Store the specimen in a desiccator, and use it within 24 h after polishing excluding the period of time during which the protective film is on it.

B.2 Specimen cleaning before the test

To wash the specimen before testing, prepare the following apparatus and perform it in an environment free from dust emissions. Then, wash the specimen in accordance with the procedure below:

a) Position the apparatus with five washing vessels and an ultrasonic washing bath (frequency: approximately 42 kHz) as shown in Figure B.1.

b) Use a laboratory beaker, made of either borosilicate glass 3.3 in accordance with ISO 3585 or quartz glass, of 200 ml or more, and fill it with a sufficient amount of washing liquid for the specimen.

c) Pour neutral detergent water solution into washing vessel No. 1. Pour distilled water into washing vessels No. 2 and No. 3. Put IPA into washing vessel No. 4.

d) Place each vessel in the ultrasonic washing bath.

e) Put IPA into washing vessel No. 5, and place it outside of the ultrasonic washing bath.

f) Adjust the temperature of the ultrasonic washing bath so that the temperature of the washing liquid for washing vessels No. 1 to 4 is 45 °C ± 3 °C.

g) Wash the specimens using the procedure described in Table B.1. The washing time for each tank shall be 30 s.

h) Wash washing vessels No. 1 to 4 in the ultrasonic washing bath by applying ultrasonic waves.

i) When transferring a specimen, do not touch the specimen's inner area (that is, the area more than 5 mm from the rim of the polished specimen face).

j) Immerse the specimen in washing vessel No. 1 (detergent), and wash the specimen. The specimen shall be shaken during washing in order to improve washing efficiency.

k) Immediately use a clean cloth to wipe off the surface of the specimen, in order to remove the remaining washing liquid. Promptly place the specimen in washing vessel No. 2.

