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Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement method of spectral transmittance of fine ceramics thin films under humid condition

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement method of spectral transmittance of fine ceramics thin films under humid condition

Céramiques techniques — Méthode de mesurage de la transmittance spectrale des films minces de céramiques fines en conditions humides

Reference number
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Contents

Page

Foreword

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The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

Introduction

Fine ceramics thin films are used in many applications such as anti-reflective coatings, infrared sensor cut filters, X-ray sensor cut filters, and band-pass filters for medical analysis equipment. Even though spectral transmittance of fine ceramics thin films are specified for each product, the refractive index and optical property change when water vapor or other vapor is adsorbed to the surface of fine ceramics thin films, because, generally, fine ceramics thin films have a columnar and voided structure. For this matter, an International Standard that could evaluate the reliability under a range of humidity was required. This International Standard provides test methods that will easily and accurately evaluate effects of humidity on optical properties of fine ceramic coatings. By establishing this International Standard, it aims for a rapid spread of this test method that will then lead to further growth of the industry.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement method of spectral transmittance of fine ceramics thin films under humid condition

1 Scope

This International Standard specifies a measurement method of spectral transmittance of fine ceramics thin films under humid condition. This International Standard provides test methods that will easily and accurately evaluate changes in optical properties of fine ceramic coatings in a humid atmosphere due to water adsorption.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9211-1, *Optics and photonics — Optical coatings — Definitions*

ISO 8980-3:2013, *Ophthalmic optics — Uncut finished spectacle lenses — Part 3: Transmittance specifications and test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8980-3 and ISO 9211-1, and the following apply.

3.1
half transmittance wavelength
$\lambda_{T1/2}$
center of a segment delimited by the wavelengths at which the transmittance is maximal and minimal in a given wavelength range

3.2
environmental mini-chamber
small chamber to keep the environmental condition of a test piece contained inside the chamber

Note 1 to entry: The chamber is equipped with optical feed-through ports with a ball valve for evacuation or air-circulation. The chamber shall be air-tight to keep the chamber in vacuum or with the atmosphere at a certain humidity by closing the ball valves after evacuation or air-circulating to expose the test piece inside to a certain atmosphere.

3.3
transmittance
fraction of intensity of incident optical beam transferring through the sample

3.4
monochromator
optical device that selects light beam with a certain wavelength with a range

3.5
spectrophotometer
optical instrument that measures spectral transmittance or reflectance

Note 1 to entry: The instrument consists of optical source, monochromator, sample chamber, optical detector, signal processor, data processor, and interface.

3.6
double beam spectrophotometer
type of spectrophotometer that is utilized to compare the light intensity between the reference sample and the test pieces

Note 1 to entry: Compared to single-beam spectrophotometer measures, a high stability of measurements is obtained. The instrument consists of optics that divide a source light beam into two beams and two detectors. The instability of a source beam mainly due to that of the source lamp is compensated by that of a reference beam.

3.7
integrating-sphere
optical component consisting of a hollow spherical cavity with its inner surface covered with a diffuse white reflective coating

Note 1 to entry: The cavity is equipped with small holes to set a sample piece and for inlet and outlet of the light beam.

4 Principle

Fine ceramics thin films sometimes possess a voided micro columnar structure, depending on film deposition conditions. If a fine ceramics thin film with a voided structure is exposed to a certain atmosphere with humidity, moisture in the atmosphere adsorbs on surfaces of the columns consisting the voided structures. The quantity of adsorbed water is affected by hydrostatic vapor pressure in the atmosphere. Depending on the quantity of water adsorbed, the refractive index of thin films changes, resulting in the change in transmittance and reflectance. To evaluate effects of water adsorption on to surface of voids of fine ceramics films on film optical properties, it is necessary to measure optical properties under a certain atmosphere, typically in an atmosphere with a high humidity. In addition to the measurements under a high humidity, for the sake of comparison, it is also necessary to measure the properties in vacuum, i.e. under the condition without water adsorption and low humidity. By comparing optical transmittance measured in vacuum, low humidity, and high humidity conditions, the stability of fine ceramics thin films shall be evaluated.

5 Ambient room environment

The test shall be carried out under an ambient laboratory room atmosphere where deviations in the temperature and humidity are small. In particular, to prevent the condensation of water in the environmental mini-chamber, after removing the mini-chamber from the humidity-temperature control chamber to the laboratory room, the ambient temperature should be controlled to a certain range. Recommended laboratory room conditions are the following:

a) temperature: 23 °C ± 2 °C;

b) relative humidity: below 70 %.

NOTE Attention is needed to dew condensation when environment temperature is low compared to that in the humidity-temperature control chamber.

6 Test piece

The test piece shall comprise a single layer or multilayer of thin films of fine ceramic deposited on to the surface of a plate or film substrate, such as glass, polymer material, with no restriction of material for the substrate if it is enough transparent in a certain range to measure transmittance of thin films. If it fits the environmental mini-chamber, in terms of size and shape, any test piece is acceptable.

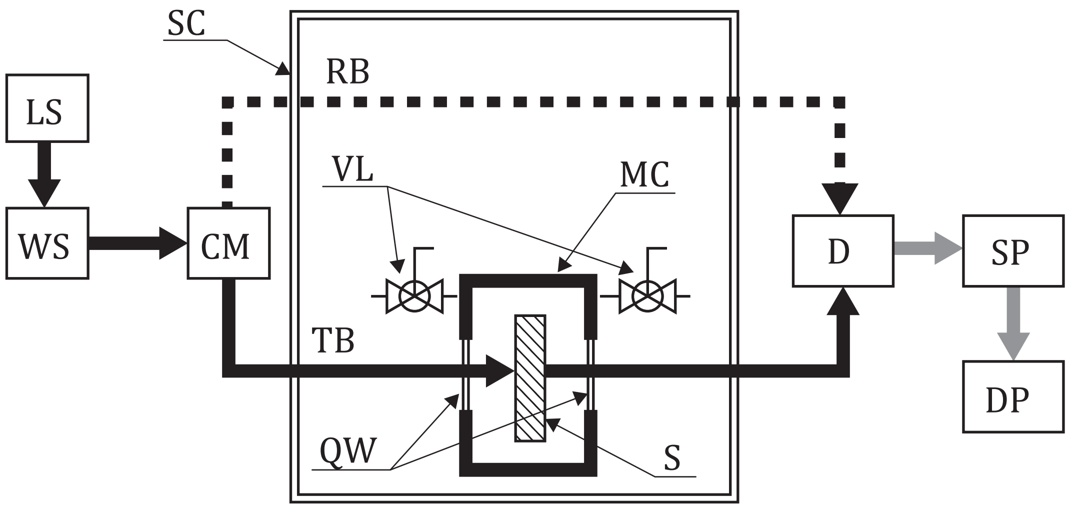
7 Measuring apparatus

7.1 Apparatus configuration

a) Spectrophotometer: Double beam method, wavelength accuracy ±0,2 nm or less at UV-visible light and ±1,0 nm or less at near-infrared range, repeatability of wavelength setting ±0,1 nm or less at UV-visible light and ±0,5 nm or less at near infrared range. For UV-visible and near-infrared range measurements, the range of 300 nm to approximately 2 500 nm is desirable. The use of the integrating sphere for photometer is recommended. In the case of measurements in a range in infrared and far-infrared, the wavelength range can be expanded by using an appropriate spectrometer.

b) Environmental mini-chamber: An airtight container which could maintain the test sample in vacuum and low- and high-humidity environments for the measurements of the spectral of each environment. Build two windows with transparent quartz glass with the sample fixed inside the chamber for the measurement of light transmittance. Also build an air inlet and exhaust ports, each with a valve. It is necessary to confirm that there is no vacuum leak from the chamber when all the valves are closed. In case of necessity, check the leak rate of the chamber by using a vacuum gauge.

The spectrometry device configuration is shown in Figure 1. Example of the blueprint is shown in Annex A.



Key

LS	light source	TB	test beam
CM	chopper mirror	RB	reference beam
WS	wavelength selector (monochrometer)	MC	environmental mini-chamber
SC	specimen compartment	VL	valve
D	detector	QW	quartz window
SP	signal processor	S	specimen
DP	data processor		

Figure 1 — device configuration for spectral transmittance measurement

c) A vacuum evacuation (exhaust) equipment: A rotary pump or a dry pump shall be used. The type does not matter only if the ultimate pressure is less than 40 Pa and if the pumping speed is larger than 1,0 l/s.

d) Temperature/humidity controlled chamber: Use types that can set the temperature at 23 °C and that can set the relative humidity range from 40 % to 80 %. It is desirable to measure the humidity around the test sample using a hygrometer.

7.2 Spectrometer calibration

Perform a wavelength and transmittance calibration for the spectrophotometer apparatus as follows

a) Wavelength calibration: The test method which uses optical glass filter, didymium, or holmium oxides shall be applied. For an infrared range, polystyrene can be used as a wavelength standard.

b) Baseline correction: Set spectral transmittance to 100 % in the range of the measuring wavelength by using the baseline adjustment function of the apparatus by measuring the transmittance of the environmental mini-chamber without setting a test piece in the chamber.

Other baseline correction methods, e.g. correction for air, are acceptable, though the method applied should be reported.

8 Environmental conditions and control procedures

Evacuate or expose the test piece to a required environmental condition in a temperature/humidity control chamber prior to optical transmittance measurements. The test piece should not be taken out from the environmental mini-chamber during a series of measurements of vacuum, low-humidity, and high-humidity conditions. Measurement environmental conditions recommended are vacuum, temperature of 23 °C ± 2 °C and R.H. of 80 % ± 2 %, and temperature of 23 °C ± 2 °C and R.H. of 40 % ± 2 % as described in the following list. If necessary, the environmental test sequence may be modified.

a) Vacuum: Evacuate the environmental mini-chamber containing a test piece inside by connecting one of its ports to a vacuum pump. Evacuate the chamber for longer than 30 min.

NOTE To reduce the amount of water remaining in thin films after the evacuation, the environmental mini-chamber may be heated up to 130 °C during evacuation. However, the mini-chamber is cooled to room temperature prior to the transmittance measurement.

b) Low-humidity environment: Place the environmental mini-chamber containing a test piece inside in the temperature/humidity controlled chamber. Open both of the valves and expose the test piece set inside the mini-chamber to a required temperature and humidity condition. The temperature and humidity of the temperature/humidity control chamber is controlled to be 23 °C ± 2 °C and 40 % ± 2 %, respectively. Retain the environmental mini-chamber in the temperature/humidity control chamber for longer than 20 min.

c) High-humidity environment: Place the environmental mini-chamber containing a test piece inside the temperature/humidity control chamber. Open both of the valves and expose the test piece set inside the mini-chamber to a required temperature and humidity condition. The temperature and humidity of the temperature/humidity control chamber is controlled to be 23 °C ± 2 °C and 80 % ± 2 %, respectively. Retain the environmental mini-chamber in the temperature/humidity control chamber for longer than 20 min.

9 Optical transmittance measurement procedure

Measure the optical transmittance in the wavelength range needed. Use the conditions of a wavelength interval of 0,2 nm, scanning speed below 120 nm/min, and fast response for a wavelength range shorter than 850 nm or the conditions of a wavelength interval of 0,2 nm, scanning speed below 150 nm/min, and fast response for a wavelength range longer than 850 nm. Other specific conditions can be applied.

a) Set up of mini-chamber: Set the mini-chamber securely to an appropriate position in the sample compartment of a spectrometer. If the mini-chamber is not securely set to a proper position, the repeatability of the measurements lowers.

b) Test piece position: Measure optical transmittance at the centre of the test piece. There is no need for optical diaphragm if the dimension of the test piece is larger than the environmental mini-chamber's window diameter.

c) Measurement of transmittance at a vacuum condition: Measure the transmittance in vacuum after an evacuation procedure. The valve has to be closed prior to cuting off the port from the vacuum pump.

d) Measurement of transmittance at a low-humidity condition: Measure the transmittance after the withdrawal of the mini-chamber from the temperature/humidity control chamber. The valves have to be closed immediately after opening the door of the temperature/humidity control chamber before withdrawing the mini-chamber from the temperature/humidity control chamber.

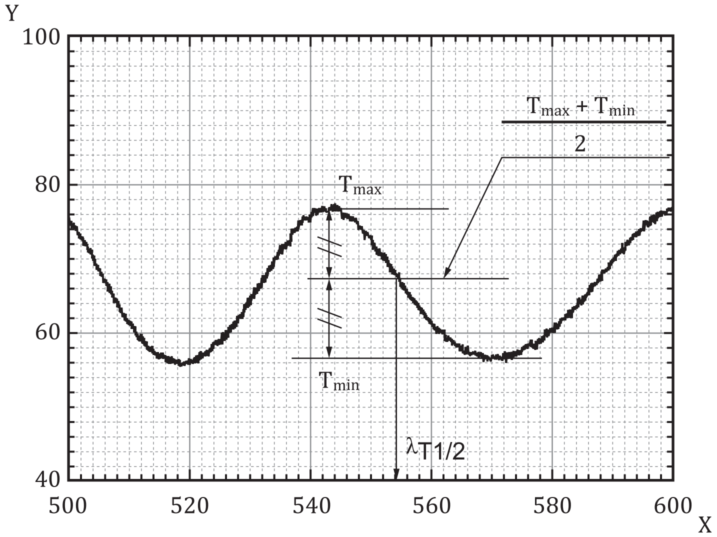
e) Measurement of transmittance at a high-humidity condition: Measure the transmittance after the withdrawal of the mini-chamber from the temperature/humidity control chamber. The valves have to be closed immediately after opening the door of the temperature/humidity control chamber before withdrawing the mini-chamber from the temperature/humidity control chamber.

10 Evaluations of measured spectra

Evaluate changes in optical transmittance of fine ceramics thin films among vacuum, low humidity, and high humidity. Evaluation methods for several typical changes observed in the spectra are shown below.

a) **Single layer films**

Obtain a half of maximum/minimum transmittance wavelength ($\lambda_{T1/2}$) from optical transmittance spectra measured under vacuum, high-humidity, and low-humidity conditions from the spectral transmittance measured. Then evaluate the shift of $\lambda_{T1/2}$ among vacuum, low-humidity, and high-humidity conditions. The use of the relative shift value is strongly recommended to evaluate the stability of thin films against water adsorption. In addition, the single layer should yield at least a set of maximum and minimum in transmittance to obtain a half maximum/minimum. If needed, the maximum or minimum in transmittance spectra may be used to evaluate stability.

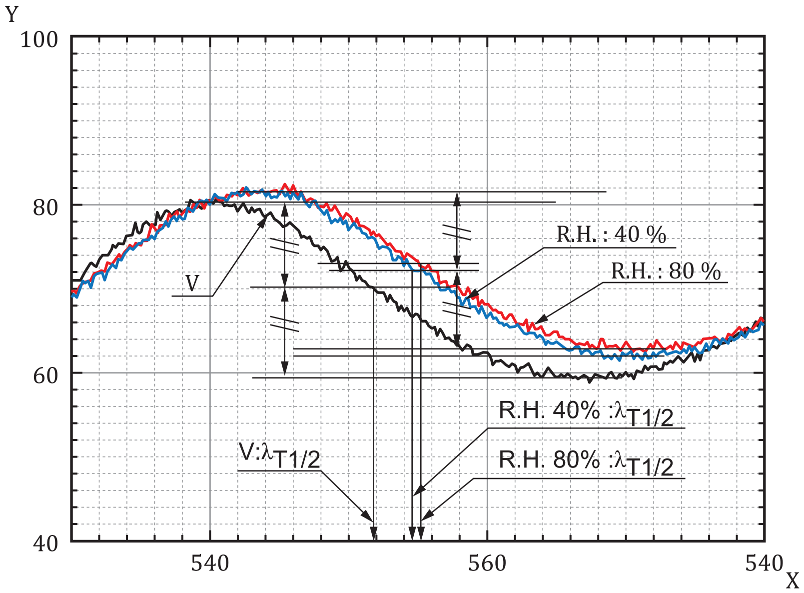


Key

X wavelength (nm)

Y transmittance (%)

Figure 2 — A half maximum/minimum transmittance wavelength ($\lambda_{T1/2}$) obtained for a single layer film



Key

X wavelength (nm)

Y transmittance (%)

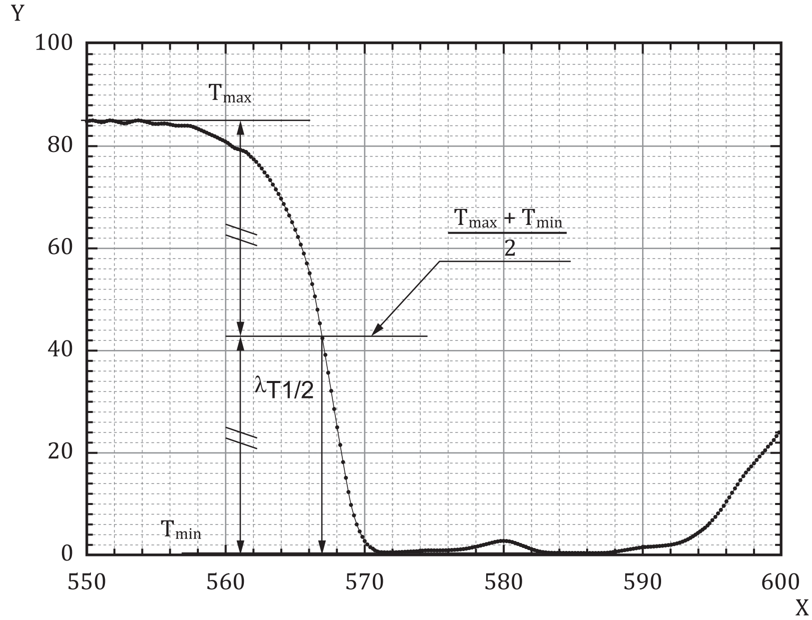
V vacuum

R.H relative humidity

Figure 3 — An example of the shift in the half maximum/minimum transmittance wavelengths (λT1/2) evaluated for a single layer thin film

b) **Edge filters**

Obtain a half of maximum/minimum transmittance wavelength at the edge wavelength ($\lambda_{T1/2}$) from optical transmittance spectra measured under vacuum, low-humidity, and high-humidity conditions. Evaluate a shift in the half of maximum/minimum transmittance wavelength ($\lambda_{T1/2}$) among vacuum, low-humidity, and high-humidity conditions. The use of the relative shift value is strongly recommended to evaluate the stability of thin films against water adsorption.

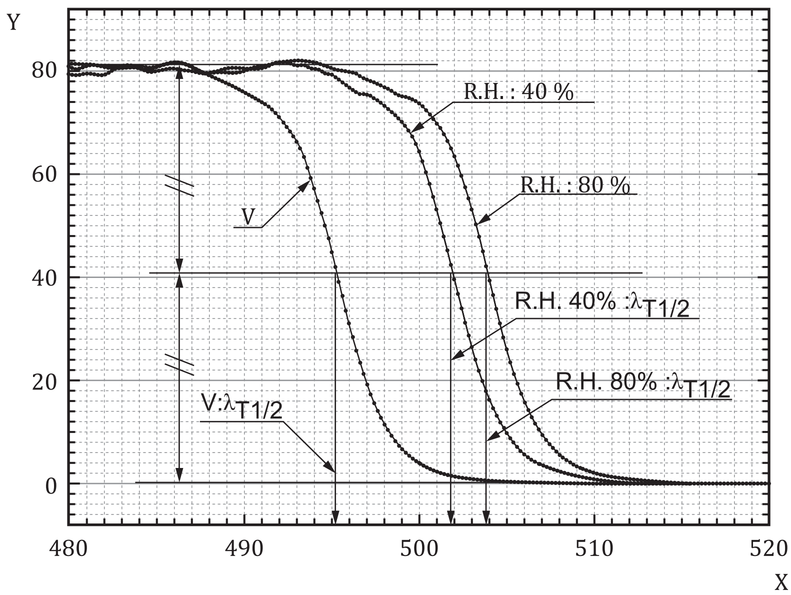


Key

X wavelength (nm)

Y transmittance (%)

Figure 4 — A half maximum/minimum transmittance wavelength ($\lambda_{T1/2}$) obtained for an edge filter



Key

X wavelength (nm)

Y transmittance (%)

V Vacuum

R.H. relative humidity

Figure 5 — An example of the shift in the half maximum/minimum transmittance wavelengths ($\lambda_{T1/2}$) evaluated for an edge filter

11 Test report

The test report shall include the following information:

a) a reference to this International Standard (i.e. ISO 17861);

b) date of the test, name of the testing establishment, temperature, relative humidity;

c) description of the test piece and its identity;

d) description of the measuring apparatus;

e) description of test conditions, vacuum evacuation time, preset humidity degree and retention time for high and low humidity environment, humidity of the surroundings of the test piece if a hygrometer is used;

f) description of measured data; if optical glass filter is used for wavelength calibration, the manufacturer and type and distribution curve and transmittance value of spectral transmittance; times taken to measurements after valves are closed for each condition;

g) spectral transmittance data for all measurements, if needed (strongly recommended to store data in digital form);

h) wavelength shift quantity of low and high humidity, a maximum or minimum transmittance and their shift in a certain range of wavelength, etc.;

i) special instruction regarding measurements.

Annex A
(informative)

Structure and function of mini-chamber

A.1 Structure

Figure A.1 indicates the blueprint of an environmental mini-chamber.

A.2 Function

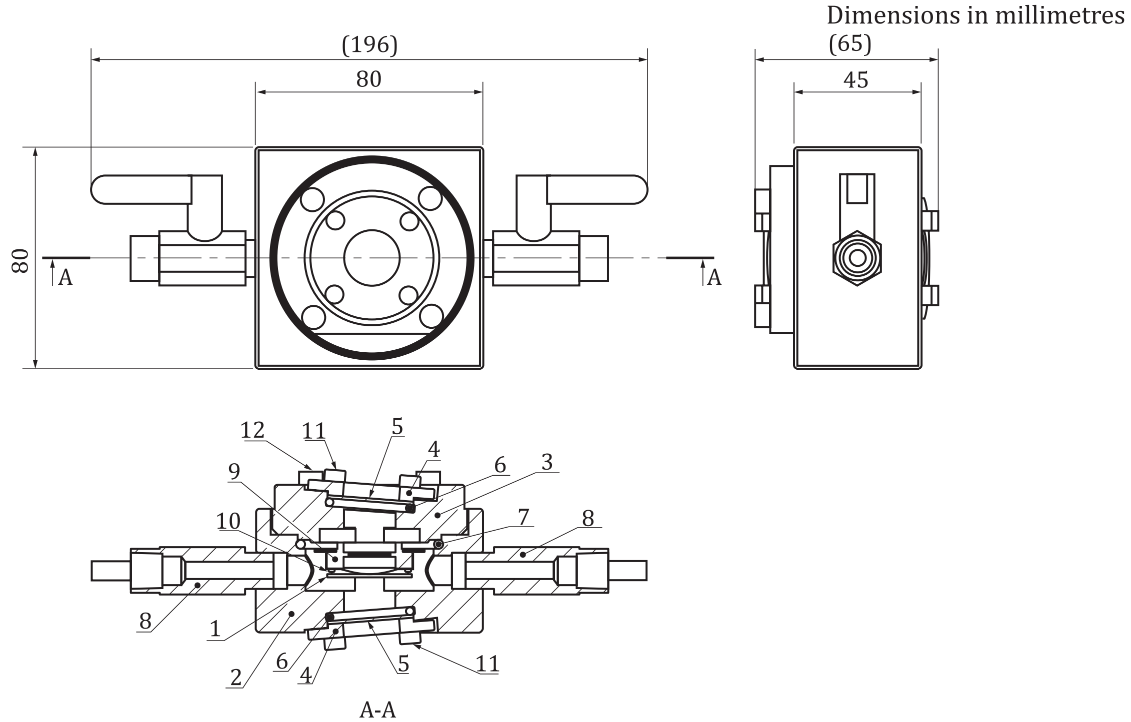
The test piece shall be fixed using wavewashers. Vacuum evacuation and installation of air with a regulated humidity shall be conducted by opening and closing the two ball valves.

A.3 Required performance

— No changes in pressure and humidity (absolute humidity) when ball valves are closed.

— Transparent windows that do not fog by humidity.

— Each window shall be fixed tilted 5° to the test piece so as not to cause optical interference.



Key

1 test piece
2 chamber (SUS303)
3 window frame (SUS303)
4 window retainer (black POM)
5 window (quartz glass)
6, 7 O- ring
8 ball valve
9 colour (PTFE)
10 wavewasher
11 M4 bolt (for fixing window retainer)
12 M5 bolt (for fixing window frame)

Figure A.1 — Blueprint of environmental mini-chamber

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