INTERNATIONAL STANDARD

ISO 14368-4

First edition
2020-08

Mineral and sapphire watch-glasses —

Part 4:
Anti-reflective treatment

Verres de montres minéraux et en saphir —

Partie 4: Traitements antireflet

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Reference number
ISO 14368-4:2020(E)

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Published in Switzerland

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This document was prepared by Technical Committee ISO/TC 114, *Horology*, Subcommittee SC 13, *Watch-glasses*.

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Introduction

Anti-reflective treatments are widely used in watch-glasses. Anti-reflective treatments are used to improve legibility of the watch dial by reducing light reflected from the watch-glasses.

When customers are wearing watches, the watches go through temperature variation, corrosion, scratch, sunlight and many other environmental conditions. The properties of the anti-reflective treatments may directly affect the appearance of the watch-glasses and the legibility of the dial, therefore this International Standard aims to clarify the test methods and the evaluations for the anti-reflective treatments.

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Mineral and sapphire watch-glasses —

Part 4:
Anti-reflective treatment

1 Scope

This document specifies the terms and definitions, the test methods and the evaluation of results of watch-glasses with anti-reflective treatments.

The document is applicable to sapphire watch-glasses with anti-reflective treatments, and it can also be used as a reference for mineral watch-glasses with anti-reflective treatments.

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 3160-2:2015, *Watch-cases and accessories — Gold alloy coverings — Part 2: Determination of fineness, thickness, corrosion resistance and adhesion*

ISO 4892-1, *Plastics — Methods of exposure to laboratory light sources — Part 1: General guidance*

ISO 4892-2, *Plastics — Methods of exposure to laboratory light sources — Part 2: Xenon-arc lamps*

ISO 9227, *Corrosion tests in artificial atmospheres — Salt spray tests*

ISO/CIE 11664-1:2019, *Colorimetry — Part 1: CIE standard colorimetric observers*

ISO 11664-2, *Colorimetry — Part 2: CIE standard illuminants*

ISO 14368-3:2003, *Mineral and sapphire watch-glasses — Part 3: Qualitative criteria and test methods*

ISO 23160:2011, *Watch cases and accessories — Tests of the resistance to wear, scratching and impacts*

CIE 15:2018, *Colorimetry*

CIE 85:1989, *Solar Spectral Irradiance*

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/

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3.1
luminous transmittance
ratio of the transmitted luminous flux to the luminous flux of the incident radiation

$$\tau_v = \frac{\int_0^\infty \tau(\lambda)\Phi_{e,\lambda}(\lambda)V(\lambda)d\lambda}{\int_0^\infty \Phi_{e,\lambda}(\lambda)V(\lambda)d\lambda}$$

where

$\tau(\lambda)$ is the spectral transmittance of the sample;

$\Phi_{e,\lambda}(\lambda)$ is the spectral radiant flux of the source;

$V(\lambda)$ is the spectral luminous efficiency;

[SOURCE: ISO 80000-7:2019, 7-31.6, modified — The definition has been slightly reworded.]

3.2
luminous reflectance
ratio of the reflected luminous flux to the luminous flux of the incident radiation

$$\rho_v = \frac{\int_0^\infty \rho(\lambda)\Phi_{e,\lambda}(\lambda)V(\lambda)d\lambda}{\int_0^\infty \Phi_{e,\lambda}(\lambda)V(\lambda)d\lambda}$$

where

$\rho(\lambda)$ is the spectral reflectance of the sample;

$\Phi_{e,\lambda}(\lambda)$ is the spectral radiant flux of the source;

$V(\lambda)$ is the spectral luminous efficiency;

[SOURCE: ISO 80000-7:2019, 7-31.4, modified — The definition has been slightly reworded.]

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4 Test methods and evaluation of results

4.1 General

The following tests are all separate tests. Except for special instructions, each test is conducted with new samples, and no superposition test is conducted. The selection of test item and test plan can be determined by agreement between the contracting parties.

After the test, the anti-reflective treatment is evaluated by visual inspection and by comparison with referenced samples. The appearance shall be checked in accordance with ISO 14368-3:2003, Annex A. The evaluations of test results are checked according to the requirements given by each test item and, if required, higher acceptance criteria of the test results should be defined by agreement between the contracting parties.

4.2 Optical characterization

The spectral distribution of standard illuminant D65 as specified in ISO 11664-2 and the luminous efficiency of the average human eye for daylight vision ($V(\lambda)=\bar{y}(\lambda)$ for 2° observer) as specified in

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ISO/CIE 11664-1:2019, 3.11, shall be used to determine the luminous transmittance τ_v or the luminous reflectance ρ_v.

The selection of the measurement geometry between transmittance and/or reflectance can be determined by agreement between the contracting parties.

4.2.1 Luminous transmittance

Parameters for the digital integration of the transmittance spectrum $\tau(\lambda)$ are: illuminant D65 and standard observer 2°, step width not exceeding 10 nm and range from 380 nm to 780 nm. The luminous transmittance factor is expressed in $\tau_v = Y$ as described in CIE 15:2018, Chapter 7.

$$\tau_v = \frac{\int_{380}^{780} \tau(\lambda) S(\lambda) \bar{y}(\lambda) d\lambda}{\int_{380}^{780} S(\lambda) \bar{y}(\lambda) d\lambda}$$

where

$S(\lambda)$ is the relative spectral power distribution of the illuminant D65;

$\bar{y}(\lambda)$ is one of the CIE colour matching function.

NOTE The measuring range between 380 nm to 780 nm can be truncated to another range (e.g. 360 nm to 740 nm) providing the result is not significantly changed (see CIE 15:2018, 7.2).

4.2.1.1 Apparatus with integration sphere

Use a spectrocolorimeter or a spectrophotometer with an integration sphere to obtain the transmittance spectrum with the Specular Component Included (SCI) according to a measuring geometry di:0°or 0°:di (see CIE 15:2018, Chapter 6).

4.2.1.2 Apparatus without integration sphere

Use a spectrocolorimeter or a spectrophotometer without integration sphere to obtain the transmittance spectrum according to a measuring geometry 0°:0° (see CIE 15:2018, Chapter 6). In this case, only the regular transmittance component is measured.

NOTE In case of a polarized light source (grating on the beam path) and due to the birefringent effect of the sapphire, it is recommended to align the optical axis of the sapphire with the polarization axis of the beam in order to minimize the fringes amplitude on the transmittance spectrum.

4.2.2 Luminous reflectance and colour

Parameters for the digital integration of the reflectance spectrum $\rho(\lambda)$ are: illuminant D65 and standard observer 2°, step width not exceeding 10 nm and range from 380 nm to 780 nm. Reflectance factor is expressed in $\rho_v = Y$ as described in CIE 15:2018, Chapter 7, and colour is expressed in $L^*a^*b^*$ values as described in CIE 15:2018, 8.2.

Use a spectrocolorimeter or a spectrophotometer with an integration sphere to obtain the reflectance spectrum in reflection mode with the Specular Component Included (SCI) according to a measuring geometry di:8°or 8°:di (CIE 15:2018, Chapter 6).

$$\rho_v = \frac{\int_{380}^{780} \rho(\lambda) S(\lambda) \bar{y}(\lambda) d\lambda}{\int_{380}^{780} S(\lambda) \bar{y}(\lambda) d\lambda}$$

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where

$S(\lambda)$ is the relative spectral power distribution of the illuminant D65;

$\bar{y}(\lambda)$ is one of the CIE colour matching function.

NOTE 1 The measuring range between 380 nm to 780 nm can be truncated to another range (e.g. 360 nm to 740 nm) providing the result is not significantly changed (see CIE 15:2018, 7.2).

In order to measure the pure reflectance, only the light reflected by the watch-glass is collected by the detector. Particularly, light beams transmitted through the watch-glass and further reflected on a back support should not be included in the signal detected (see Figure A.2). It might be necessary to check if other light sources (ambient light, reflection on various support material or setting surface for example) do not affect the measurement in a significant manner.

NOTE 2 When colour parameters are measured, other colour spaces can be used such as L^*C^*h, xyY or $L^*u^*v^*$ (according to CIE 15:2018, Chapter 8).

4.2.3 Evaluation of results

Acceptance criteria of the test results should be defined by agreement between the contracting parties. Reference values of luminous transmittance, luminous reflectance and colour are given in Annex A.

4.3 Adhesive force

4.3.1 Test method

Use an adhesive tape with a peel adhesion of 2,9 N/cm to 3,3 N/cm. The tape is adhered to the treated surface, ensuring that bubbles between the tape and the treated surface are eliminated. After 10 s, tear the tape quickly with the force perpendicular to the treated surface. The tape used shall not leave any glue residues on the treated surface.

4.3.2 Evaluation of results

After the test, the anti-reflective treatment shall show no chap, bubble, delamination and peel-off.

4.4 Humidity test

4.4.1 Test method

The watch-glasses with anti-reflective treatments are placed in the constant temperature and humidity equipment. The temperature inside the chamber is set to 40 °C, with a tolerance of ±2 °C. The relative humidity inside the chamber is set to 93 %, with a tolerance of ±5 %. The test is carried out for at least 48 h.

4.4.2 Evaluation of results

After the test, the anti-reflective treatment shall show no chap, bubble, delamination and peel-off.

4.5 Thermal shock test

4.5.1 Test method

Place the samples in a thermal chamber stabilized at 70 °C ± 2 °C without humidity contribution during 2 h. Then soak them immediately in deionized water at 5 °C ± 2 °C during 30 s minimum. Repeat this cycle 5 times minimum. Samples shall be dried after each cycle.

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4.5.2 Evaluation of results

After the test, the anti-reflective treatment shall show no chap, bubble, delamination and peel-off.

4.6 Salt spray test

4.6.1 Test method

According to ISO 9227, neutral salt spray test shall be carried out for at least 24 h. Then the adhesive force test specified in 4.3 is carried out.

4.6.2 Evaluation of results

After the test, the anti-reflective treatment shall show no chap, bubble, delamination, peel-off and obvious (dis)coloration.

4.7 Synthetic sweat test

4.7.1 Test method

According to ISO 3160-2:2015, 7.4, synthetic sweat test shall be carried out for at least 24 h. Then the adhesive force test specified in 4.3 is carried out.

4.7.2 Evaluation of results

After the test, the anti-reflective treatment shall show no chap, bubble, delamination, peel-off and obvious (dis)coloration.

4.8 Abrasion resistance

4.8.1 Sample preparation

The test samples, with flat surface, are made of the same material and the same anti-reflective treatment, and are produced using the same method as the watch-glass.

4.8.2 Test method

The samples (treated surface up) are cleaned and fixed on the friction testing machine. The friction head moves back and forth along the same path, and the distance between the two ends is twice the diameter of the friction head. After 100 times reciprocating friction, clean and check the samples by visual inspection. The friction head is a rubber whose diameter is 6,5 mm to 7,0 mm, and hardness is (75 ± 5) IRHD, with 6 to 8 layers dry degreasing cloth outside. The perpendicular stress applied on the friction head is (5 ± 1) N. The average rate of the test is (1 ± 0,1) cycle per second.

4.8.3 Evaluation of results

After the test, the anti-reflective treatment shall show no obvious scratch or wear.

4.9 Scratch resistance

4.9.1 Test method

The test method shall be in accordance with ISO 23160:2011, 5.2, 5.3 and 5.4.

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4.9.2 Evaluation of results

After the test, the anti-reflective treatment shall show no obvious scratch.

4.10 Sunlight resistance

4.10.1 Test method

4.10.1.1 Equipment

The test equipment used shall be made of a closed chamber provided with test specimen holders and one or more light sources correctly filtered, such as a Xenon arc lamp or metallic halide lamp emitting a radiance with a spectrum close to the one of the sun at sea level, in accordance with CIE 85:1989, Table 4. Filtering should minimize irradiance under 290 nm and above 800 nm.

An airflow sweeping the test specimens shall be used to control the temperature.

The test specimen holder is made to ensure a uniform irradiance on all sample surfaces, with a ±10 % tolerance on the irradiance fixed between 290 nm and 400 nm. If necessary, a rotating test specimen holder device will help meet this prescription.

This equipment is equipped with devices which allow the measurement of irradiance on test specimens and also of the temperature and humidity in the chamber. Irradiance shall be regulated between 290 nm and 400 nm. A temperature and humidity regulation is recommended.

The irradiance measuring device as well as the black standard thermometer used for the measurement of the maximum temperature attainable by samples shall comply with ISO 4892-1.

The test equipment can be equipped with devices for sample immersion or watering cycles. The deionized water used during the immersion or watering cycles shall be pure enough (conductivity <5 µS/cm, <1 µl/l[1] of solid material) to avoid stains on the test specimens.

4.10.1.2 Test conditions

According to the specificity of horological external parts, the following conditions are recommended for the full duration of the test:

a) irradiance between 290 nm and 400 nm at sample level: 60 W/m^2 maximum;

b) exposure time: 36 h minimum;

c) black standard temperature (BST according to ISO 4892-1): 65 °C maximum;

d) relative humidity: 60 % ± 5 %;

e) The minimum energy dose in the 290 nm to 400 nm range shall be 7,8 MJ/m^2.

NOTE Energetic dose [J/m^2] = Irradiance [W/m^2] × Exposure duration[s].

Test temperature and humidity significantly influence the ageing speed of the exposed sample. The lamps may have different distribution of wavelengths in their spectrum depending on their type, their age or the type of reflecting environment surrounding samples. For these reasons, differences in results may appear between tests performed in different facilities or in the same installation using different test parameters. In the context of real exposures, differences are also observed.

Different test conditions, such as irradiance, temperature indicated by the black standard thermometer, relative humidity and test duration shall be mutually agreed upon between the stakeholders.

Should watering cycles be required, reference shall be done to ISO 4892-2.

1) 1 µl/l = 1 ppm.

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Examples of parameters depending on the chosen lamp are given in Annex B.

4.10.1.3 Operating procedure

a) The samples to be tested are laid flat on the test specimen holder, the surface to be tested is oriented towards the luminous source.

b) Unless there is a referenced sample perfectly identical to the samples to be tested, a part of the surfaces to be tested is hidden with an aluminium foil or any equivalent solution.

c) A periodic examination of samples can be carried out during the test. In that case, results shall be mentioned in the test report.

d) During the examination, evaluate the evolution by comparing with the referenced sample or the unexposed surface.

e) Place the sample on a background offering a great contrast with its colour. It shall be illuminated above the control surface with a white light of a higher than 500 lx intensity, close to natural light. A standard D65 lamp of an intensity of more than 500 lx shall be used when arbitrations are necessary.

f) The colour (or its changes) shall be determined by spectrocolorimetric measurement, in accordance with CIE 15.

g) The results are expressed in change of colour related/associated to an energetic dose rather than the duration of exposure.

h) The test report shall indicate in detail the type of luminous source used as well as all the applied parameters [at the minimum those described in 4.10.1.2 a) to e)].

Light source(s) shall be replaced when the specified irradiance cannot be reached anymore.

4.10.2 Evaluation of results

After the test, the anti-reflective treatment shall show no chap, bubble, delamination, peel-off and obvious (dis)coloration.

4.11 Cleaning test

4.11.1 Test method

Impregnate the gauze with ethyl alcohol. Push the gauze against the anti-reflective surface with an indicative pressure ranging from (4 to 8) N/cm^2, and wipe it back and forth 3 times.

4.11.2 Evaluation of results

After the test, the anti-reflective treatment shall show no chap, bubble, delamination, peel-off and obvious (dis)coloration.

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Annex A
(informative)

Optical characterization of watch-glasses

A.1 Luminous transmittance

The luminous transmittance values, τ_v, of watch-glasses are described in Table A.1. The values without treatment refer to flat, parallel and polished surfaces of a pure monocrystalline Al_2O_3 sapphire or a mineral glass.

Table A.1 — Luminous transmittance, τ_v

Classification	Luminous transmittance (without treatment) %	Luminous transmittance (with treatment) %	Added luminous transmittance value[a] %
sapphire watch-glasses	85 to 87	one-side treatment ≥88 double-side treatment ≥91	≥3 ≥6
mineral watch-glasses	~91	≥91,5	≥0,5

NOTE The transmittance values in the table are reference values. The values can be different because of treatment material, treatment technology and so on. The specified values are defined by agreement between the contracting parties.

[a] Added luminous transmittance value is the difference between the luminous transmittance of watch-glasses after treatment and the luminous transmittance before treatment.

A.2 Luminous reflectance and colour

The luminous reflectance, ρ_v, of a pure monocrystalline Al_2O_3 sapphire watch-glass with flat, parallel and polished surfaces, without treatment is comprised between 13 % to 15 %.

The colour shows a L^* value of approximately 45 and a^* and b^* values close to zero, with no significant influence of the watch-glass' thickness.

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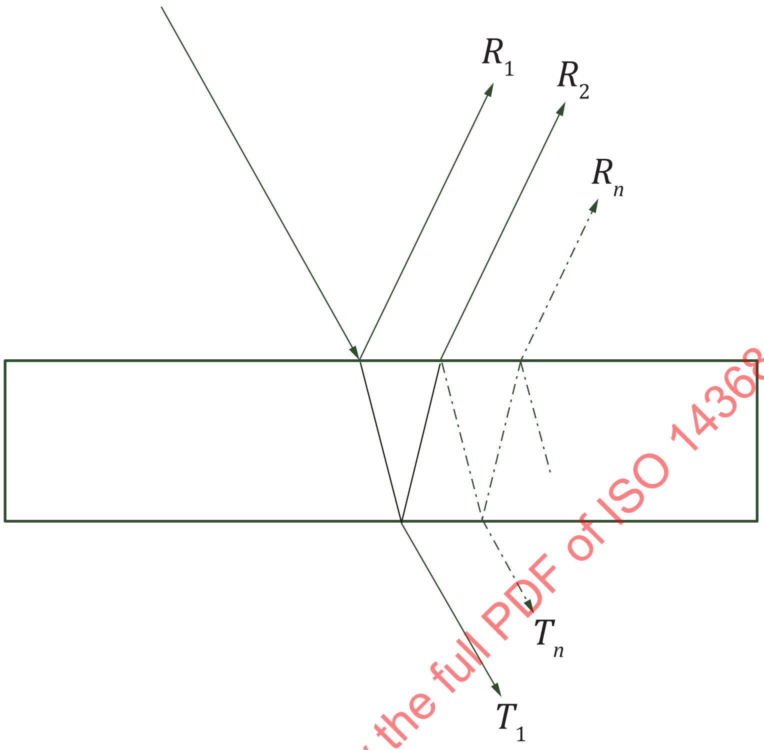
A.3 Drawings

A.3.1 Light paths through watch-glass



Key

R_1 reflectance from the top interface air/glass
R_2 reflectance from the bottom interface glass/air
R_n multiple reflectance inside the watch-glass
T_1 transmittance component
T_n multiple transmittance components

Figure A.1 — Light paths through watch-glass

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Fresnel equations applied on the 2 interfaces of the watch-glass explains that each interface contributes ~50 % to the total reflectance depending on the refractive index *n* of the material (see Figure A.1).

This means that the bottom interface also contributes to the reflectance.

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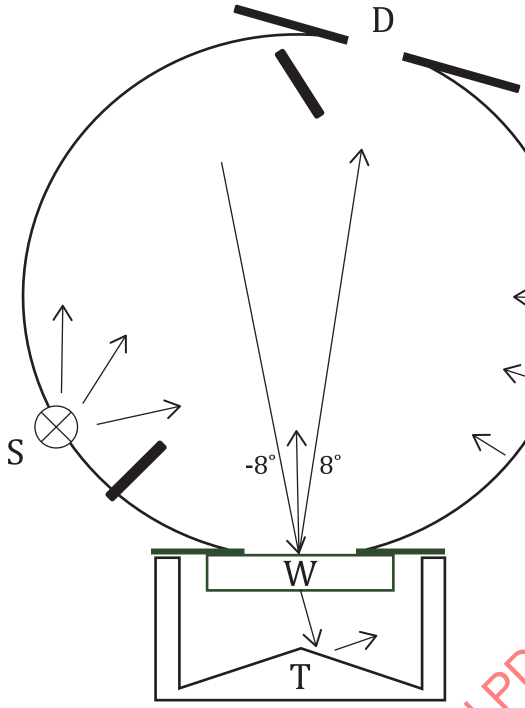
A.3.2 Reflectance measurement using integration sphere



Key

D detector

S light source

T light trap

W watch-glass

Figure A.2 — Reflectance measurement using integration sphere

The light source illuminates the sphere covered with a white material showing Lambertian reflectance which illuminates the sample.

The reflected beam is collected by the detector.

The transmitted beam through the sample should not be allowed to return into the sphere: for this purpose, it shall be directed far away from the apparatus or trapped to avoid any extra contribution in the measurement of the reflectance components by the detector.

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