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**Rubber, vulcanized — Resistance to weathering —**

**Part 3:**  
Methods of exposure to artificial light

**iTeh STANDARD PREVIEW**

*Caoutchouc vulcanisé — Résistance aux intempéries —*

*Partie 3: Méthodes d'exposition à la lumière artificielle*

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## Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4665-3 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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# Rubber, vulcanized — Resistance to weathering —

## Part 3:

### Methods of exposure to artificial light

#### 0 Introduction

The effects of light on the colour and other properties of rubbers is of considerable technical and commercial importance. Methods of exposing rubbers to natural light are dealt with in part 2 of this International Standard.

Testing with laboratory light sources usually has one of the following purposes:

- a) to obtain results by accelerated testing under controlled conditions to indicate behaviour which would be given by prolonged exposure to natural daylight;
- b) for control tests on a material of known light resistance to establish that the level of quality of different batches does not vary from a known acceptable control.

NOTE — Guidance concerning the problems of correlation between the effects of exposure to artificial light sources and those obtained after exposure to natural light is given in annex A.

For some specific applications, it may be possible to use certain fluorescent lamps as light sources. However, there is at present insufficient information on the reliability and repeatability of the results from such exposures to warrant inclusion of this type of apparatus in this part of ISO 4665. Investigations into these matters are being undertaken and it is hoped that, as a result, fluorescent lamp light sources may be incorporated at a future revision. Notes giving some existing information on fluorescent lamps and their possible use are given in annex D which may assist interested parties in making their own investigations into the use of such devices.

#### 1 Scope and field of application

This part of ISO 4665 specifies methods of exposing vulcanized rubbers to an artificial light source in order to assess changes produced by such exposure.

Of the different light sources available, the xenon arc is advantageous in that it can, when correctly filtered and maintained, yield a spectrum most closely approximating to that of daylight. Hence, this part of ISO 4665 is confined to this light source.

This part of ISO 4665 also specifies means of determining radiation dosage.

#### NOTES

- 1 For the method of determination of changes in properties after exposure, see ISO 4665-1.
- 2 Tests described in this part of ISO 4665 are usually carried out in the absence of any stress applied to the test pieces. A stress or strain may, however, be applied by agreement between the interested parties, provided that its use is fully reported.

#### 2 References

ISO 105, *Textiles — Tests for colour fastness*

— Section A02: *Grey scale for assessing change in colour;*

— Section B01: *Colour fastness to light: Daylight.*

ISO 1826, *Rubber, vulcanized — Time-interval between vulcanization and testing — Specification.*

ISO 4661-1, *Rubber, vulcanized — Preparation of samples and test pieces — Part 1: Physical tests.*

ISO 4665, *Rubber, vulcanized — Resistance to weathering*

— Part 1: *Assessment of changes in properties after exposure to natural weathering or artificial light.*

— Part 2: *Methods of exposure to natural weathering.*

#### 3 Principle

Test pieces are exposed to the light source together with means of assessing the light dosage. These means may comprise one or more of the following:

- a) physical standards which change in colour or other well-defined properties upon exposure to light, the degree of change indicating the light dosage;
- b) instrumental means of measuring irradiance and/or integrating this to give the light dosage over a period of time.

For indication of behaviour that might be found under natural daylight, changes in the test pieces are determined at each of a number of light exposure stages, to give a sufficiently full picture of the performance throughout exposure. This method is also used, with interpolation if necessary, to find the amount of exposure needed to produce a specified change in the material.

For control tests, a suitable exposure stage is selected in advance from a knowledge of the expected light resistance of the material and the change in the test piece is evaluated at this stage only. Alternatively, the exposure stage may be determined at which a defined change in the properties of the exposed test pieces occurs.

## 4 Apparatus

### 4.1 Laboratory light sources

#### 4.1.1 General

To improve the correlation between results of exposures to laboratory light sources and natural light, it is necessary that the spectrum of the laboratory light source be as near as possible to that of the natural light, particularly in the ultra-violet region, because some rubbers are very sensitive to the spectral distribution of the radiation. However, the intensity of the radiation will generally be higher than that of natural sunlight because of the need to accelerate the degradation processes in laboratory tests, even though correlation with the natural exposure condition may be weakened (see annex A).

Recommendations for the integrated irradiance and spectral distribution of simulated solar radiation for test purposes in the laboratory are given in CIE Publication No. 20 (TC-22), 1972<sup>1)</sup>. The recommendation for the integrated irradiance for testing the deterioration resistance of materials and equipment exposed to natural radiation is  $1\ 000 \pm 100\ \text{W/m}^2$ .

The spectral distribution described in CIE Publication No. 20 (TC-22), 1972, is reproduced in annex C.

NOTE — For various reasons it is not practicable to reproduce this radiation level exactly in either spectral distribution or intensity with presently available apparatus.

#### 4.1.2 Xenon arc lamp

The xenon arc lamp emits radiation in a range which extends from below 270 nm in the ultra-violet through the visible spectrum and into the infra-red. For exposure tests, light from the lamp is filtered to reduce shorter wavelength emissions and also to remove as much of the infra-red as possible, so that radiation reaching test pieces exposed to it has a spectral power distribution that closely matches sunlight. The facility may also be available to reduce short wavelength energy further so that an alternative spectrum, similar to that of solar radiation as received behind window glass, may be obtained. These two modes of operation are often available on the same equipment, using different filter systems.

The irradiance at the test piece face in the wavelength range 300 to 890 nm shall normally be  $1\ 000 \pm 200\ \text{W/m}^2$ . If, exceptionally, other intensities are used, these shall be stated in the test report. Irradiance below 300 nm shall not exceed  $1\ \text{W/m}^2$ . The irradiance shall not vary by more than  $\pm 10\%$  over the whole test piece area.

The characteristics of xenon arcs and filters are subject to changes in use due to ageing and these items shall be replaced at appropriate intervals. Further, they are subject to changes due to the accumulation of dirt and they shall be cleaned at appropriate intervals.

### 4.2 Test enclosure

**WARNING** — Care shall be taken to protect laboratory staff from the effects of ozone.

The enclosure contains a cylindrical frame carrying test piece holders, with provision for passing air over the test pieces for control of temperature. Accumulation of any ozone generated by the lamps shall be prevented either by venting the air to the outside of the building or by other appropriate means.

The lamp shall be so placed that the amount of radiation received by the test pieces does not vary by more than  $\pm 10\%$  over the entire area in which the test pieces are exposed.

To reduce the effect of any eccentricity in the lamp, or when more than one lamp is used in a single enclosure to increase the amount of radiation, the radiation distribution shall be improved by rotating the frame carrying the test pieces around the light source and, if necessary, by periodically changing the position of each test piece vertically.

The test piece holders may rotate on their own axes as well as rotating with the frame, thus exposing to the direct radiation of the light source the side of the test piece holder that was previously in the dark. This method helps to maintain a low black panel temperature on the test piece. Alternatively, dark cycles may be produced by cycling the source on and off. If either of these cycles is used, it shall be fully reported.

**4.3 Black panel thermometer**, to indicate the test temperature, consisting of a blackened absorbing metal plate that approximates the absorption characteristics of a "black body". The plate shall be at least 1 mm thick and of a size to fit the test piece holders. The temperature of the plate is indicated by a suitable thermometer or thermocouple making good thermal contact.

The black panel thermometer shall be mounted in a test piece holder with the blackened metal side facing the lamp and readings are taken after sufficient time for the temperature to become steady.

The black panel temperature is controlled by adjustment of the cooling air circulation.

NOTE — This control can conveniently be achieved by means of a thermostat whose sensor is placed in the test enclosure. When it is necessary to minimize variation of temperature to within  $\pm 1\ ^\circ\text{C}$ , care must be taken to place the sensor in the best position so that it responds to the temperature variation as fast as possible.

1) Published by the International Commission on Illumination, Bureau Central, 52, boulevard Malesherbes, F-75008 PARIS, France.

**4.4 Wet and dry bulb thermometers or other suitable instruments**, inserted into the test space and shielded from the lamp radiation, for measuring the relative humidity of the air passing over the test pieces, which may be controlled at an agreed value, if required.

**4.5 Spray**, if required, for spraying the test pieces with distilled or deionized water. The spray system shall be made from inert materials which do not contaminate the water.

#### 4.6 Test piece holders

Test piece holders may be in the form of an open frame, leaving the back of the test piece exposed, or they may provide the test piece with a solid backing. They shall be made from inert materials, for example aluminium or stainless steel. Brass, steel or copper shall not be used in the vicinity of the test pieces.

The backing used may affect the results and therefore shall be fully reported.

#### 4.7 Means of determining radiation dosage

One of the following is required, depending upon the method selected.

**4.7.1 Blue dyed wool standards No. 1 to No. 7**, as specified in ISO 105, section B01 and the gray scale for assessing change in colour as specified in ISO 105, section A02 (see also annex E, clauses E.1 and E.2).

**4.7.2 Other physical standards** agreed between the interested parties.

**4.7.3 Instrumental means of measuring light dosage**, comprising a photoreceptor system mounted beside the test pieces and connected to an integrating device to indicate the total energy received over a period.

The photoreceptor system shall be sensitive to radiation received over a solid angle similar to that over which radiation is received by the test pieces. The spectral response of the photoreceptor system shall correspond to the spectral regions that produce changes in the test pieces.

The instrument shall be calibrated in suitable radiometric units, such as joules per square metre for the specific light source. The calibration shall not be affected by variations in light intensity or temperature.

#### NOTES

1 Research is proceeding in certain countries on the spectral response required to give the best estimate of light dosage in relation to its effect on materials. It is known that for some materials the short-wave end of the ultra-violet range is particularly important, but it is not possible at present to recommend a particular spectral response.

2 Prolonged exposure of an instrumental measuring device to radiation will probably affect its reliability, and therefore periodic checks should be carried out.

3 For the physical standards of light dosage (blue wool) the spectral response is determined by the choice of the particular dyestuffs used.

**4.8 Apparatus to assess changes in properties**, as specified in ISO 4665-1 or in the relevant product or material specification.

## 5 Test pieces

### 5.1 General

As far as possible, each test piece shall be cut from freshly moulded sheet, or, if required, from a finished product in accordance with ISO 4661-1. It shall have an undamaged test surface. Only test pieces of similar dimensions and having approximately the same exposed area shall be compared.

### 5.2 Test pieces for determination of changes in colour

Use rectangular strips the surface dimensions of which are at least 15 mm and compatible with the particular apparatus used for the exposure (see clause 4).

Two test pieces shall be used for a test of a single exposure stage. In other cases, the total number of test pieces required will be determined by the number of exposure stages, but this number shall be at least two. It may be necessary to increase the number for products where the colour is not uniform or the sensitivity to exposure is irregular. A further test piece shall be stored in the dark at standard laboratory temperature and shall constitute the reference standard for assessment of colour change.

NOTE — It is known that some materials will change colour during storage in the dark. In this case, the change in colour of the reference test pieces shall be reported.

### 5.3 Test pieces for determination of changes in other properties

The dimensions of the test piece shall normally be those specified in the appropriate test method for the property or properties to be measured after exposure. For some tests, the exposed test piece may also be in the form of a sheet or other shape from which test pieces can later be cut for specific tests.

The total number of test pieces required will be determined by the number of exposure stages. Additional test pieces shall be stored in the dark at standard laboratory temperature, and shall constitute the unexposed controls for the determination of the property after each exposure stage.

#### NOTES

1 The dimensions and number of test pieces will also depend on the particular apparatus used for the exposure (see clause 4).

2 Test results obtained from test pieces prepared before exposure may differ from those obtained from test pieces cut from exposed sheet or products owing to the possible effect of the exposure on the cut edges.

### 5.4 Storage and conditioning

For all test purposes, the minimum time between vulcanization and testing shall be 16 h in accordance with ISO 1826.



For non-product tests, the maximum time between vulcanization and testing shall be 4 weeks and for evaluations intended to be comparable the tests, as far as possible, should be carried out after the same time-interval.

For product tests, whenever possible, the time between vulcanization and testing should not exceed 3 months. In other cases, tests shall be made within 2 months of the date of receipt of the product by the customer.

Test sheets and test pieces shall not, at any time, be allowed to come into contact with those of a different composition. This is necessary in order to prevent additives which may affect resistance to weathering, such as antioxidants, from migrating from one vulcanizate into adjacent vulcanizates.

## 6 Test conditions

### 6.1 Black panel temperature

The preferred black panel temperature is  $55 \pm 3$  °C. Other temperatures may be used according to the nature of the material and its proposed application, but it should be recognized that high black panel temperatures will increase the tendency for thermal degradation effects to influence the test results.

NOTE — The black panel temperature represents the highest test piece surface temperature likely to be achieved. Test pieces of lighter colours and thinner test pieces, where some cooling from the back occurs, will have lower temperatures.

### 6.2 Relative humidity

The preferred relative humidity is  $(65 \pm 5)$  %. Other values may be used according to the nature of the material and its proposed application. When another value is agreed, it should be one of the following:  $(35 \pm 5)$  %,  $(50 \pm 5)$  % or  $(90 \pm 5)$  %.

#### NOTES

1 Owing to the varying temperatures of test pieces having different colours and thicknesses the moisture content of the air very close to the test pieces cannot be taken to correspond to the relative humidity of the air as measured.

2 The relative humidity will not be maintained if the spray (6.3) is used.

### 6.3 Spray

If required, test pieces may be sprayed with distilled or deionized water intermittently under specified conditions.

If the spray is used, the maximum temperature recommendations of 6.1 apply to the end of the dry period.

The spray cycle shall be selected from the list given in table 1. Unless otherwise agreed, the standard spray cycle for general use shall be that with a 18 min spray time and 102 min dry interval.

NOTE — The other cycles are included in recognition of their use in some countries.

Table 1 — Spray cycles

Time of spraying	Dry interval between spraying
min	min
3	17
5	25
12	48
18	102

## 7 Procedure

### 7.1 Mounting of test pieces

Attach the test pieces (clause 5) to the holders (4.6) in the equipment in such a manner that the test pieces are not subjected to any applied stress.

Identify each test piece by suitable indelible marking, but not on the areas to be used in testing. As a check, a plan of the mounting positions may be retained. Expose the blue dyed wool standards (4.7.1) or other physical standards (4.7.2) in a similar manner to the test pieces for determination of exposure stage.

Alternatively, suitably mount instrumental means of measuring light dosage (4.7.3) so that the photoreceptor measures the irradiance level at the test piece location. (But see note 2 to 4.7.3.)

If water spray is used (see 4.5), protect blue dyed wool standards from the water by a suitable transparent cover, for example silica or polymethylmethacrylate sheet containing no ultra-violet absorber. It is advisable first to check that the covers are transparent to the incident light by running a comparison test on covered and uncovered standards under dry conditions.

If desired, in the case of test pieces used to determine change in colour and appearance, a portion of each test piece may be shielded by an opaque cover throughout the test. This gives an unexposed area adjacent to the exposed area for comparison. This is useful for checking the progress of exposure, but the data reported shall always be based on the contrast with the unexposed comparison pieces.

### 7.2 Exposure to light sources

Bring the test conditions in the test enclosure (4.2) for the specified light source (see 4.1) to the required levels (see clause 6). Maintain these conditions throughout the exposure.

Expose the mounted test pieces, and if required the appropriate light standards, for the specified period of exposure stage. Although the test piece frame may be rotated around the lamp, it is desirable, in those cases where test pieces are stacked, that vertical position of the test pieces be varied from time to time to reduce further any local inequalities of exposure. When test pieces are so adjusted, they shall not be inverted in any direction.

If it is necessary to remove a test piece for periodic inspection, care shall be taken not to handle or disturb the test surface. After inspection, the test piece shall be returned to its holder or

to the test enclosure with its test surface in the same direction as previously. This procedure may not be required when both sides of the test piece are freely exposed in a holder capable of rotating on its own axis (see 4.2).

### 7.3 Measurement of radiation dosage

#### 7.3.1 General

Measurements of radiation dosage may only be compared for like light sources. Misleading conclusions may result if measurements of unlike sources, for example artificial light and natural light or different types of artificial light, are compared.

#### 7.3.2 Using blue dyed wool standards (ISO 105)

Details are given in annex B.

NOTE — The blue dyed wool standards developed for textile testing have been used in the testing of rubbers. It is well recognized that this method has severe limitations, particularly when consecutive exposures of standard No. 7 are used. Consecutive exposure of standard No. 7 should only be employed when no better alternative is available.

#### 7.3.3 Using other physical standards

Measure as appropriate to the standard material and as agreed between the interested parties.

#### 7.3.4 Using instrumental means

When using instrumental determination of radiation dosage, the exposure stage is given in terms of the amount of energy received by the instrument and test pieces.

### 7.4 Determination of changes after exposure

These shall be determined as specified in ISO 4665-1.

## 8 Test report

The test report shall contain the following information:

- a) sample details
  - 1) a full description of the sample and its origin,
  - 2) compound details, cure time and temperature, where appropriate,
  - 3) method of preparation of test piece;
- b) test method (the reference of this part of ISO 4665);
- c) test details
  - 1) specific details of the lamp used and, if possible, the irradiance at the surface of the test piece,
  - 2) mode of operation of lamp and filter system used,
  - 3) black panel temperature used,
  - 4) relative humidity of air passing over the test pieces,
  - 5) conditions of water spray, if used,
  - 6) nature of backing, support and attachment, if used,
  - 7) conditions of test piece rotation, if used,
  - 8) procedures for determining exposure stages, including method used to determine radiation dosage, if used (if instrumental methods are used, the exposure stage should be expressed in joules per square metre),
  - 9) applied stress on test piece, if used;
- d) test results
  - 1) exposure stages used,
  - 2) presentation of results as required by ISO 4665-1,
  - 3) the changes in the comparison test piece, if determined;
- e) the date of test.

## Annex A

# Correlation of the effects of exposure to artificial light sources and exposure to natural daylight

(This annex forms an integral part of the Standard.)

### A.1 General

The quality and intensity of solar radiation at the earth's surface vary with climate, location and time, but the average of a whole year's weathering at a particular location normally differs little from one year to another.

In the case of natural weathering, there are factors other than solar radiation which affect the ageing process, such as temperature, temperature cycling, humidity, ozone, etc.

Many rubber products are exposed under a static or cyclic tensile strain during service, and with these the cracking caused by ozone attack is often a more serious consequence of natural weathering than ageing caused by light. It should be noted that the test pieces used in this part of ISO 4665 are normally free of strain when exposed to the artificial light source.

Experience has shown that correlation between results of testing with laboratory light sources, and in natural daylight at a particular locality, is imprecise and can only be assumed for a specific type and formulation of a material, and for particular properties, where it has been demonstrated from past experience.

With different rubbers the correlation factor for the same laboratory source may be different.

### A.2 Factors tending to decrease the degree of correlation

#### A.2.1 Use of ultra-violet radiation of wavelengths shorter than those occurring in natural exposure

Such radiation has only a small effect on natural exposure because of its absence or low intensity at ground level.

The xenon arc lamp, when appropriately filtered, produces radiation with a spectral energy distribution similar to that of average sunlight. By way of comparison, carbon arc lamps emit radiation high in ultra-violet light content, open-flame types producing considerable amounts of radiation shorter in wavelength than that found in natural daylight. Fluorescent tubes can be selected to have a spectral output corresponding to that of the ultra-violet region in sunlight.

#### A.2.2 Use of high test temperatures, particularly with materials which can readily undergo changes from thermal effects alone

In such cases, the results may indicate the effects of heating rather than those of light exposure.

The xenon arc lamp produces a large proportion of infra-red radiation that should be reduced by filters. Efficient cooling of the test pieces is necessary to guard against overheating. Emission of infra-red radiation is also a feature of carbon arc lamps. In contrast, fluorescent tube sources produce little infra-red radiation, and there is generally no problem with overheating of the test pieces.

#### A.2.3 Use of a spectral distribution of radiation that differs widely from that of daylight

In this respect, the correctly filtered xenon arc is a reasonably satisfactory source for laboratory testing. By contrast, the enclosed carbon arc and the open-flame carbon arc both give excess radiation in the region between 350 and 420 nm, relative to sunlight. Fluorescent tube sources normally are deficient in visible radiation relative to ultra-violet radiation as compared with sunlight.

#### A.2.4 Factors that accelerate the rate of change in the test pieces

In general, factors that accelerate the rate of change in the test piece also tend to reduce the degree of correlation with natural daylight. These include the use of a very high light flux or very thin test pieces.

It may be possible to establish a relationship between the laboratory light dosage and the natural dosage to give similar effects with particular materials, but with others the correlation for the same laboratory source may be different.

### A.3 Control testing

In control testing, the above factors also apply, but the situation is often more favourable than when attempting to forecast the behaviour of a material in natural light, for the following reasons:

- a) The behaviour of the material will already be well known.



b) The object of the test is restricted to showing whether or not the light resistance of the test piece is less than that of the standard product.

c) A test piece representative of a product having reduced light resistance in daylight, due to some error of formulation or manufacture, is likely also to give poorer results under laboratory sources and this may be sufficient for a control test without needing close correlation.

d) Reproducibility should be improved when testing is carried out with the same apparatus, using the same cycle and the same duration of exposure. It is recommended that for control testing the method should be closely specified and results reported as required by this part of ISO 4665 (see clause 8).

Nevertheless, variation in ultra-violet spectral distribution, and high temperatures in particular, may give completely misleading results even in control testing, and should not be used except in cases where a satisfactory correlation has been established for the particular product concerned.

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