INTERNATIONAL STANDARD

ISO 4665

Third edition
2016-01-15

Rubber, vulcanized or thermoplastic — Resistance to weathering

Caoutchouc vulcanisé ou thermoplastique — Résistance aux intempéries

Reference number
ISO 4665:2016(E)

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Foreword

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The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This third edition cancels and replaces the second edition (ISO 4665:2006), which has been technically revised with the following changes:

— normative references have been updated, small editorial changes made for clarification and compression set added to mechanical properties that could be measured.

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Introduction

A number of different exposure techniques can be used to provide information on the effects of environmental stresses such as light, heat, and water on rubbers. Each of these has its own particular application and relevance. Explanation of, and guidance on, methods for exposure to natural and artificial weathering is given in ISO 877-1 and ISO 4892-1. Particular guidance on exposure to determine resistance to ozone is given in ISO 1431-1. The methods for exposure to weathering standardized for plastic materials are essentially suitable for rubbers, and hence this International Standard refers to the relevant ISO standards for plastics for the apparatus and procedures.

It is desirable that the procedures for the determination of changes in properties are the same whatever exposure is used and that the results should be expressed in a uniform manner. Such procedures are specified in this International Standard.

Exposure to weathering alters the properties of the material, particularly in the surface layer. The test method used to determine changes in properties should be selected after consideration of the properties of the material which are important in its proposed application and taking into account the fact that degradation might be concentrated at the surface layer. The methods chosen ought to be capable of measuring change in properties with sufficient precision within the ranges which are important in practice, so as to provide significant criteria of change.

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Rubber, vulcanized or thermoplastic — Resistance to weathering

1 Scope

This International Standard specifies methods for the exposure of vulcanized or thermoplastic rubbers to natural or artificial weathering and methods for the determination of changes in colour, appearance, and physical properties resulting from exposure.

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 105-A02, *Textiles — Tests for colour fastness — Part A02: Grey scale for assessing change in colour*

ISO 877-1:2009, *Plastics — Methods of exposure to solar radiation — Part 1: General guidance*

ISO 877-2:2009, *Plastics — Methods of exposure to solar radiation — Part 2: Direct weathering and exposure behind window glass*

ISO 877-3:2009, *Plastics — Methods of exposure to solar radiation — Part 3: Intensified weathering using concentrated solar radiation*

ISO 1431-1, *Rubber, vulcanized or thermoplastic — Resistance to ozone cracking — Part 1: Static and dynamic strain testing*

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 4892-3, *Plastics — Methods of exposure to laboratory light sources — Part 3: Fluorescent UV lamps*

ISO 4892-4, *Plastics — Methods of exposure to laboratory light sources — Part 4: Open-flame carbon-arc lamps*

ISO 18314-1, *Analytical colorimetry — Part 1: Practical colour measurement*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 877 and ISO 4892 and the following apply.

3.1
control
material exposed alongside the test material for comparison

Note 1 to entry: The control, for example, may be a material of similar or related composition to the test material or a material having a known response to the exposure conditions.

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3.2
file test piece
portion of the material to be tested which is stored under conditions in which it is stable and is used for comparison between the exposed and the original state

3.3
masked area
portion of the exposed test piece which is protected from light exposure by masking

3.4
weathering reference material
reference material whose weathering degradation properties are well documented and repeatable when exposed to identical conditions

3.5
exposure stage
interval of exposure between determinations of change in properties, expressed as time or radiant exposure

4 Principle

Test pieces are exposed to natural or artificial weathering and the resulting changes in colour, appearance, and selected physical properties are determined.

5 Exposure to direct weathering, to weathering using glass-filtered daylight or to intensified weathering by daylight using Fresnel mirrors

Carry out the exposure in accordance with the relevant method of ISO 877, with the following additions and modifications:

— For tests under strain, prepare the test pieces and place them under strain in accordance with ISO 1431-1.

— It is recommended that one or more of the weathering reference materials described in Annex A be used as a control, the material selected depending on the type of rubber compound under test. The effects of natural weathering on these materials in a range of climates are detailed in Reference [1] and Reference [2].

— Condition the test pieces in accordance with ISO 23529.

6 Exposure to laboratory light sources

Carry out the exposure in accordance with ISO 4892-1 and, as relevant, ISO 4892-2, ISO 4892-3, or ISO 4892-4, with the following additions and modifications:

For tests under strain, prepare the test pieces and place them under strain in accordance with ISO 1431-1.

It is recommended that one or more of the weathering reference materials described in Annex A be used as a control, the material selected depending on the type of rubber compound under test. The effects of natural weathering on these materials in a range of climates are detailed in Reference [1] and Reference [2].

Condition the test pieces in accordance with ISO 23529.

NOTE 1 The general guidance on weathering of plastics given in ISO 4892-1 is applicable to rubbers.

NOTE 2 In general, an open-flame carbon-arc lamp is not recommended because it is not representative of sunlight.

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7 Changes in colour

7.1 Apparatus

7.1.1 Instrumental assessment

Use a colour or colour change apparatus meeting the requirements described in ISO 18314-1.

7.1.2 Visual assessment

Use the grey scale in accordance with ISO 105-A02.

7.2 Test piece

The test piece shall be in any form which allows sufficient flat area for the determination of colour change.

NOTE It is convenient to use a test piece required for the determination of change in physical properties.

7.3 Procedure

7.3.1 General

The choice of instrumental or visual assessment of colour change shall be made by agreement between the interested parties.

7.3.2 Instrumental assessment

Measure the colour of the test piece before and after each exposure stage in general accordance with ISO 18314-1. If required, also measure the colour of a file test piece or a masked area.

7.3.3 Visual assessment

Compare the contrast rating of the exposed test piece and an unexposed file test piece, and if required, a masked area, after each exposure stage, in accordance with the procedure given in ISO 105-A02.

8 Changes in other appearance properties

Examine each test piece visually after each exposure stage for changes in appearance using, if appropriate, the procedure given in a relevant International Standard. Some examples of parameters used to assess change in appearance are listed in Annex B.

Ozone cracking shall be assessed in accordance with ISO 1431-1.

NOTE Cracking or crazing in rubber test pieces exposed outdoors can result from light ageing as well as from ozone attack. A distinction is not always possible, particularly with light-coloured rubbers. Sunlight crazing is characterized by shallow cracks and usually occurs independently of strain, whereas a threshold strain has to be exceeded for ozone cracking to occur. Where there is doubt, it is useful to expose an unstrained test piece alongside the strained test piece for comparison.

9 Changes in physical properties

9.1 General

The properties measured may be those specified in an International Standard for the material or as agreed between the interested parties. Some suitable properties are listed in Annex B.

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9.2 Apparatus

Apparatus in accordance with the relevant International Standard for the determination of the properties shall be used.

9.3 Test pieces

Test pieces shall comply with the requirements of the relevant International Standard for the determination of the properties chosen.

Test pieces may be cut from an exposed sheet of material. In such cases, the test pieces shall be taken not less than 20 mm from any fixtures holding the material or from supports that are not intended to simulate the conditions of exposure of the material in service. In no circumstances shall any of the material be removed from the exposed face during test piece preparation.

9.4 Procedure

Condition the test pieces and carry out the property determination in accordance with the procedure given in the relevant International Standard. Determine the initial properties on unexposed test pieces and the properties of exposed test pieces after each exposure stage. If required, also determine the properties of file test pieces or masked areas.

Repeat the measurements carried out on the sample test pieces on test pieces from any control being used.

NOTE With some tests, the results will depend on which side of the test piece is exposed. For example, in tests involving bending or flexure, the result depends on whether the exposed or unexposed surface is placed under tension.

10 Expression of results

10.1 Change in colour

10.1.1 Instrumental measurements

Determine the colour difference in accordance with ISO 18314-1.

10.1.2 Visual measurements

Record the contrast rating of the test pieces which have been compared. If the observed contrast lies between two ratings on the grey scale, record an intermediate rating. For example, a rating of 3-4 signifies that the contrast is greater than 3 but less than 4.

Also, record the type of colour change using the following terms:

Hue	More blue or less blue
	More green or less green
	More red or less red
	More yellow or less yellow
Purity	Brighter
	Duller
Lightness	Lighter
	Darker

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A typical report of colour change by visual assessment would be: “More yellow, duller, lighter, grey scale 2-3”.

10.2 Changes in other appearance properties

For quantitative methods, calculate the change in property from:

$$C = P - P_x \quad (1)$$

where

P is the initial property;

P_x is the property at exposure stage x.

For qualitative methods, express the result on a scale agreed between the interested parties. The following is recommended for properties other than ozone cracking:

0 none

1 barely perceptible

2 moderate

3 substantial

NOTE This scale is arbitrary and, although it is of considerable value when assessing several test pieces at the same time, great care is necessary in interpreting results from different observations.

Surface cracking on test pieces exposed under strain shall be assessed for resistance to ozone in accordance with ISO 1431-1.

10.3 Changes in physical properties

Calculate the result of each measurement in accordance with the test method in the relevant International Standard. The change in property from the initial or file test piece value, P, to the post-exposure value, P_x, may be expressed in one of the following ways:

a) as a percentage of the initial or file value:

$$\frac{P_x}{P} \times 100$$

b) as the change in the property:

$$P - P_x$$

c) the change as a percentage of the initial or file value:

$$\frac{P - P_x}{P} \times 100$$

d) as a plot of the property against the time of exposure or the radiant exposure.

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11 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 4665;

b) the sample details:

   1) a full description of the sample and its origin,

   2) details of the compound and the cure conditions, where appropriate,

   3) the method of preparation of the test pieces,

   4) details of the use of file test pieces or masked areas, if relevant, and

   5) details of any weathering reference material or other control used;

c) the test details:

   1) references to the relevant test method standards for determination of the changes in properties,

   2) the type of exposure and the apparatus used,

   3) the location and details of the exposure site for natural weathering, if relevant,

   4) a complete description of the exposure cycle or exposure conditions used,

   5) the procedure used to determine the exposure stages,

   6) whether the test pieces were placed under strain and, if so, the degree of strain,

   7) the nature of any backing, support or attachments used,

   8) the conditions of storage of the file test pieces, if relevant,

   9) the method used to determine the radiant exposure, if relevant,

   10) details of washing, if relevant, and

   11) any deviations from the standard procedures;

d) the test results:

   1) the exposure stages, expressed as time or radiant exposure,

   2) the climatic data and/or radiant exposure,

   3) the colour changes, if relevant,

   4) other changes in appearance, if relevant,

   5) the resistance to cracking in the case of test pieces exposed under strain,

   6) the individual values obtained for each property measured,

   7) the test result at each exposure stage for each property measured, and

   8) the change in each property measured and the units in which the property is expressed;

e) the date of the test.

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Annex A
(informative)

Weathering reference materials

A.1 General

This annex describes weathering reference materials which may be used for preparing control test pieces for assessing changes in physical properties in natural and artificial weathering.

A.2 Types of rubber

Four types of compound, using SBR, EPDM, CR, and NR, are given in Table A.1 to Table A.4.

In a correlation study conducted by Japanese experts, these four rubbers showed superior performance as weathering reference materials. In particular, SBR gave the most linear relationship between exposure and the changes in various properties.

It was also observed in the study that the changes in 100 % modulus and elongation at break showed a gradual change, even in a three-year outdoor exposure.

Consequently, it was found that there was a significant correlation between outdoor exposure testing and exposure testing with laboratory light sources for these four rubbers.

Other compounds were also studied (see Reference [1]) but are not recommended. Carbon black compounds without antioxidants showed a large change after a three-year outdoor exposure, but only a small change with laboratory light sources. Non-black compounds showed a large performance change in the early stages of weathering tests, but did not give suitable results in long-term exposures.

A.3 Composition

The compositions of these compounds are shown in Table A.1 to Table A.4.

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Table A.1 — Composition of SBR compound

Ingredients	Parts by mass
SBR1502	100
Carbon black, N330	50
Zinc oxide	3
Stearic acid	1
Antioxidant, 6PPD[a]	2
Antioxidant, TMQ[b]	2
Wax[c]	1
Accelerator, TBBS[d]	1
Sulfur	1,75
Vulcanization: 160 °C, 25 min	

[a] *N*-1,3-dimethylbutyl-*N'*-phenyl-*p*-phenylenediamine.

[b] Polymerized 2,2,4-trimethyl-1,2-dihydroquinoline.

[c] Melting point: 65 °C; density: 0,93 Mg/m^3.

[d] *N*-tert-butylbenzothiazole-2-sulfenamide.

Table A.2 — Composition of EPDM compound

Ingredients	Parts by mass
EPDM[a]	100
Carbon black, N330	80
Zinc oxide	5
Stearic acid	1
Paraffinic process oil[b]	50
Antioxidant,TMQ[c]	2
Accelerator, TMTD[d]	1
Accelerator, MBT[e]	0,5
Sulfur	1,5
Vulcanization: 160 °C, 20 min	

[a] EP24 [Mooney viscosity ML1+4 (100 °C): 65; ethylene content (mass %): 54; ENB termonomer content (mass %): 4,5] from JSR Corporation, or equivalent.

[b] Diana PW90 process oil [viscosity (98,9 °C): 11,25 cSt; pour point: –15 °C; aniline point: 127,7 °C; density: 0,87 Mg/m^3] from Idemitsu Kosan Corporation, or equivalent.

[c] Polymerized 2,2,4-trimethyl-1,2-dihydroquinoline.

[d] Tetramethylthiuram disulfide.

[e] 2-Mercaptobenzothiazole.

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Table A.3 — Composition of CR compound

Ingredients	Parts by mass
CR (sulfur-modified type) [a]	100
Carbon black, N330	25
Zinc oxide	5
Magnesium oxide	4
Stearic acid	0,5
Antioxidant, 6PPD[b]	2
Vulcanization: 160 °C, 15 min	

[a] Skyprene R22 from Tosoh Corporation or equivalent.

[b] *N*-1,3-dimethylbutyl-*N'*-phenyl-*p*-phenylenediamine.

Table A.4 — Composition of NR compound

Ingredients	Parts by mass
NR (RSS 1)	100
Carbon black, N330	35
Zinc oxide	5
Stearic acid	2
Antioxidant, 6PPD[a]	2
Antioxidant,TMQ[b]	2
Wax[c]	1
Accelerator, TBBS[d]	0,7
Sulfur	2,25
Vulcanization: 150 °C, 10 min	

[a] *N*-1,3-dimethylbutyl-*N'*-phenyl-*p*-phenylenediamine.

[b] Polymerized 2,2,4-trimethyl-1,2-dihydroquinoline.

[c] Melting point: 65 °C; density: 0,93 Mg/m^3.

[d] *N*-tert-butylbenzothiazole-2-sulfenamide.

A.4 Use of weathering reference materials

One or more of the four types of rubber specified in the tables should be used, preferably based on the same polymer as the material under test. If this is not possible, the use of the SBR compound is recommended.

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Annex B
(informative)

Some properties which can be determinedto assess change after exposure

B.1 Appearance

— colour

— gloss

— chalking

— cracks, crazes, pits, holes, or porous appearance

— growth of microorganisms

— migration of materials from the interior to the surface

B.2 Mechanical properties

— tensile stress-strain properties

— dynamic modulus and loss factor

— hardness

— tear strength

— tension set

— compression set

— stress relaxation

B.3 Other properties

— dimensions

— resistivity

— electric breakdown strength

— permittivity

— chemical analysis

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[1] Aimura Y., & Wada N. Reference Materials for Weathering Tests on Rubber Products. Polym. Test. 2006, **25** (2) pp. 166–175

[2] Mitsuhashi K., Okutsu S., Takane Y. The Study of Weathering Reference Materials for Rubber. Materiaru Raifu Gakkaishi. 2003, **15** (1) p. 15 [in Japanese]

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ICS 83.060

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