TC45

International Standard ISO 4665/2

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION●МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ●ORGANISATION INTERNATIONALE DE NORMALISATION

Rubber, vulcanized — Resistance to weathering — Part 2: Methods of exposure to natural weathering

Caoutchouc vulcanisé — Résistance aux intempéries — Partie 2: Méthodes d'exposition aux agents atmosphériques

First edition — 1985-09-15

UDC 678.063 : 620.193.2 **Ref. No. ISO 4665/2-1985 (E)**

Descriptors : rubber, vulcanized rubber, tests, environmental tests, determination, weather resistance.

ISO 4665/2-1985 (E)

Price based on 8 pages

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.

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 4665/2 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

Printed in Switzerland

Rubber, vulcanized — Resistance to weathering — Part 2: Methods of exposure to natural weathering

0 Introduction

Weathering tests of the type specified in this part of ISO 4665 are needed to evaluate the performance of materials intended for outdoor use. The results of such tests should be regarded only as an indication of the effect of weathering. Results obtained at one time may not be strictly comparable with those obtained at another, although, in general, samples exposed for any period of several years and examined at the same time of year will show comparable behaviour. Even in long-term tests, the results may be affected by the season in which the tests are started.

The results of short-term exposure tests can give an indication of the relative outdoor performance but should not be used to predict the absolute long-term performance of a material. The results of tests carried out for less than 12 months will depend on the particular season of the year in which they are commenced.

It should be noted that the method of exposure is usually designed to expose the material to the most severe effects associated with any particular climate. It should therefore be borne in mind that the severity of exposure in actual use is, in most cases, likely to be less than that specified in this part of ISO 4665, and allowance should be made accordingly when interpreting the results. For example vertical exposure is considerably less severe in its effect on rubbers, particularly in tropical regions, where the sun is at a high angle when it is most powerful, and also because rain-water drains more rapidly from test pieces.

In the northern hemisphere, north-facing surfaces are likely to be less severely degraded than south-facing ones because they are less exposed to solar radiation, but the fact that they may remain wet for longer periods may be of significance for materials that are affected by moisture.

1 Scope and field of application

This part of ISO 4665 specifies methods of exposing vulcanized rubbers to natural weathering in order to assess changes produced after specified stages of such exposure.

It includes procedures designed for estimating the resistance of rubber to ozone cracking under atmospheric conditions of exposure.

NOTE — For the method of determination of changes in properties after exposure, see ISO 4665/1.

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 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1431/1, *Rubber, vulcanized — Resistance to ozone cracking — Part 1: Static strain test.*

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

ISO 4661, *Rubber — Preparation of test pieces.*

ISO 4665/1, *Rubber, vulcanized — Resistance to weathering — Part 1: Assessment of changes in properties after exposure to natural weathering or artificial light.*

3 Principle

Exposure outdoors to natural daylight of test pieces, or if required sheets and other shapes from which test pieces can later be cut. After a given exposure stage, removal for the determination of changes in colour, appearance or mechanical properties. The exposure stage may be a given interval of time or expressed in terms of a given solar radiation dosage. The latter is preferred whenever the main objective of the exposure is to determine resistance to light ageing since it minimizes the effect of variations in the quality and intensity of solar radiation with climate, location and time.

Unless otherwise specified, test pieces for the determination of change in colour and change in mechanical properties are exposed in an unstrained state. If required they may be exposed under window glass. Test pieces for the determination of ozone resistance are exposed under one or more tensile strains and are examined for cracking at certain intervals. Ozone resistance is expressed according to the procedures given in ISO 1431/1.

Climatic conditions and variations during the test are monitored and reported with other conditions of exposure.

4 Apparatus

4.1 Exposure equipment, consisting essentially of a test rack on to which test pieces may be mounted directly or in suitable holders.

The rack, holders and other fixtures shall be made from inert materials that will not affect the test results. Wood, non-corrosive aluminium alloy, stainless steel or ceramics have been found suitable. Materials having thermal properties which differ from these may give different results.

Brass, steel or copper shall not be used in the vicinity of the test pieces. Where test pieces are to be exposed in the strained condition, the apparatus shall be made of a material which does not readily decompose ozone (aluminium is a suitable material).

The design of the rack shall be suited to the types of test pieces being tested, but for many purposes a flat frame mounted on a support is suitable. This frame shall consist of rails of wood or other approved materials to which the test pieces may be secured. When installed, the rack shall be capable of giving the desired angle of inclination (see 6.1) and shall be such that no portion of the test pieces shall be closer than 0,5 m to the ground or any other obstruction.

For tests under strain, the test piece holder shall secure the test piece at the required elongation. In the case of unstrained test pieces, fixtures shall be secure but should apply as little stress as possible to the test pieces and permit shrinkage, expansion or warping to occur without constraint, so far as possible.

When backing is necessary to support the test pieces, it shall be of an inert material. Also it shall be so designed that there is no pronounced sagging of the test piece, but it shall be able to support the test piece with a minimum of contact with its underside, to prevent retention of rain-water. In such cases, fine strand wire netting may be used.

If exposure under glass is required, the rack shall be placed in a suitable open-bottomed box covered with a framed lid of glass. The lid should have an unobstructed area greater than that of the rack to avoid shading. It is also recommended that there is sufficient space between the lid and the rack to ensure adequate ventilation; 75 mm has been found suitable.

The glass used for the lid shall be flat, uniformly transparent and without defects. For most purposes, a glass of 3 mm thickness, having a transmittance of approximately 90 % from 370 to 380 nm in the visible range of the spectrum, and a transmittance of less than 1 % at 300 to 310 nm and shorter wavelengths, is recommended. To maintain these characteristics it is usually necessary to replace the glass at intervals of not more than 2 years.

NOTE — Exposure under glass may give rise to different results compared to exposure to the open atmosphere because of a difference between under glass and open air temperature.

4.2 Apparatus for measurement of climatic factors, appropriate to the method adopted.

NOTE — See 7.2 for equipment required for the determination of radiation dosage.

5 Test piece

5.1 Form and preparation

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.

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. It shall have an undamaged test surface, and, when used to measure ozone resistance, shall not have a test surface that has been cut or buffed.

Only test pieces of similar dimensions and having approximately the same exposed areas shall be compared.

5.2 Number of test pieces

The number of test pieces for each test condition or exposure stage shall be that specified in the appropriate test method for the property or properties to be measured after exposure.

The total number of test pieces required will be determined by the number of exposure stages.

5.3 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 test, 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.

For tests intended to determine the resistance of rubber to ozone cracking, test pieces may be conditioned in the strained state prior to exposure. This is often desirable in order to allow the development of the protective wax bloom before exposure. In such cases, after stretching the test pieces to the desired test strain (see 6.3), the test pieces shall be conditioned for a period of between 48 and 96 h in an essentially ozone-free atmosphere in the dark; the temperature for this conditioning shall normally be the standard laboratory temperature (see ISO 471), but other temperatures may be used if appropriate for particular applications. The test pieces shall not be touched or otherwise disturbed in any way during the conditioning period. For tests intended to be comparable, the conditioning time and temperature shall be the same.

6 Test conditions

6.1 Exposure aspect

Preferably, the exposed surface of the material or test pieces shall be at 45° to the horizontal facing the equator. Other aspects may be required for particular sites or for special purposes, for example vertical exposure with any specified aspect may be required to reproduce the conditions on the outside of buildings. Exposure at 10° to the horizontal facing the equator approximates to horizontal exposure and permits drainage of excess rain-water.

To obtain maximum annual irradiation of the test piece, the angle to the horizontal of the plane of the test piece shall be the exact latitude angle of the site.

6.2 Exposure site

To obtain the most reliable information, weathering trials should be conducted at sites in a number of different environments, in particular those that compare as closely as possible with the intended conditions of use. For guidance on climatic conditions see 9.3.

Normally, the site shall be on open ground well away from trees and buildings. For exposure at 45° facing south, no obstruction, including adjacent racks, in an easterly, southerly or westerly direction shall subtend a vertical angle greater than 20°, or in a northerly direction greater than 45°. For exposures in the southern hemisphere facing north, corresponding provisions apply.

Unless service conditions dictate otherwise, natural soil covering is recommended, for example grass in temperate regions or sand in desert regions. Vegetable growth shall be kept cut low.

Additionally, for some applications, it may be desirable to include exposure in uncleared areas in jungle or forest in order to assess the effects of biological growth, termites and rotting vegetation. In choosing such sites, care shall be taken to ensure that

a) the uncleared site is truly representative of the general environment;

b) the exposure facilities and access paths do not grossly interfere with or modify the environment.

6.3 Conditions for test pieces

For the determination of changes in colour and in mechanical properties, test pieces shall be exposed in the unstrained state unless otherwise specified. The test surface shall normally be exposed to the open atmosphere, but if required the test pieces may be exposed to light under glass (see 4.1).

For the determination of resistance to ozone cracking, test pieces shall be exposed under static tensile strain. Such tests should normally be carried out at one or more of the following elongations:

5 ± 1 %	10 ± 1 %	15 ± 2 %
20 ± 2 %	30 ± 2 %	40 ± 2 %
50 ± 2 %	60 ± 2 %	80 ± 2 %

For all tests, test pieces shall normally be exposed without backing, the rear surface being open to the air. Backing (see 4.1) may be required to simulate conditions of use or to support thin films. The effect of backing may be highly significant through its influence on reflectance and heat absorption, and the sample exposed must be considered to consist of test piece plus backing.

NOTE — In cases where the intended use of the material renders it necessary to consider exposure in contact with specific backing materials, the test may be modified to take account of this requirement.

7 Exposure stages

The exposure stages at which changes in properties of the test piece are determined are specified by one of the following methods.

NOTE — The same exposure stage (by whichever method it is defined) will not necessarily give the same changes in a test piece irrespective of the site of exposure. The exposure stages specified are regarded as giving only a general indication of the degree of exposure, and results should always be considered in terms of the characteristics of the exposure site as well.

7.1 Exposure time

The stages shall be specified in terms of the duration of exposure selected from the following, unless otherwise specified:

weeks: 1; 4; 16; 28

years : 1; 1,5; 2; 3; 4; 6

NOTE — The results for exposure stages of less than 1 year will depend on the season of the year in which the exposure was made. In the longer exposure stages, seasonal effects are averaged but results may still depend on the particular season in which exposure was started (for example whether started in Spring or Autumn).

7.2 Radiation dosage

Since solar radiation can be an important factor in the deterioration of rubbers during weathering, exposure stages may be defined in terms of the amount of radiation received by the test pieces. Note that this measurement is not suitable for those cases where strained test pieces are used to assess resistance to ozone cracking.

If required, radiation dosage shall be measured by use of the following methods.

NOTE — Measurement of solar radiation does not take into account the influence of rain, temperature, ozone and other atmospheric pollutants on the weathering of rubbers. The effects these have on blue dyed wool standards for example will not be the same as that on rubber. Note also that light is not required when ozone attacks rubbers (see annex C, clause C.3).

7.2.1 Blue dyed wool standards 1 to 7 as specified in ISO 105-B01 exposed under a suitable transparent protective cover (see the note), and the **grey scale**, specified in ISO 105-A02 (see annex A).

NOTE — It is advisable to check that the covers are transparent to the incident light by running a comparison test on covered and uncovered standards under dry conditions.

7.2.2 Other physical standards, agreed between the parties concerned.

7.2.3 Photoreceptor system, mounted beside the test pieces and connected to an integrating device to indicate the total energy received over a period.

This measurement includes a considerable amount of energy in the infra-red region which has no direct photochemical effect in the weathering of rubber, though it does affect the temperature of the test piece. It may therefore be preferred to confine the measurement to the wavelength ranges which are photochemically active, mainly the ultraviolet, and, to some extent, visible light wavelengths. As an example, radiation of wavelengths between 300 and 780 nm may be measured.

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 and of known spectral response agreed between the interested parties. 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.

8 Procedure

8.1 Mounting of test pieces

Attach the test pieces (clause 5) to the test rack (4.1). 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 shall be retained.

If desired a portion of each test piece may be covered 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 test pieces.

8.2 Determination of change in colour or appearance and in mechanical properties

Expose the test piece for the appropriate exposure stage or stages, then remove from the rack and determine the changes in accordance with ISO 4665/1 and the appropriate test methods. If the exposure stage is to be expressed in terms of radiation dosage, the appropriate means of determinating solar radiation (see 7.2) shall be mounted either in a similar manner to the test pieces or adjacent to the test pieces.

Normally test pieces should not be artificially washed during exposure but if washing is required in special circumstances, it shall be carried out with distilled water or water of equivalent purity, and great care shall be taken to avoid abrasion or other damage to the weathered surface.

Regular inspection and maintenance at the site is required for refixing loose test pieces and repairing damage or deterioration to equipment, particularly after storms. If a glass lid is used over the rack, this shall be kept clean during the test.

8.3 Determination of resistance to cracking

Expose test pieces at one or more of the elongations specified in 6.3 and examine them periodically for cracking by means of a lens of magnification about X 7, following the procedures of ISO 1431/1. The test pieces shall not be handled or bumped when carrying out the examination.

Three alternative procedures for exposure of test are permissible.

8.3.1 Procedure A

Strain the test pieces at 20 % elongation and examine them for development of cracking after a specified exposure stage.

8.3.2 Procedure B

Strain the test pieces at one or more of the elongations given in 6.3. If only one elongation is used, this shall be 20 % unless otherwise specified. Note the time until the first appearance of cracks at each elongation.

8.3.3 Procedure C

Strain the test pieces at not fewer than four of the elongations given in 6.3. Note the time until first appearance of cracks at each elongation so that the threshold strain can be estimated.

NOTES

1 These three procedures are based on those given in ISO 1431/1 for the determination of ozone resistance under laboratory test conditions, to which reference should be made for general guidance on ozone testing.

2 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 in light-coloured (non-black) rubbers. Sunlight crazing is characterized by shallow cracks and usually occurs independently of strain, whereas a threshold strain must 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.

8.4 Climatic observations

Throughout all tests record climatic conditions and changes that may affect the result (see 9.3).

9 Expression of results

9.1 Determination of changes in colour and in mechanical properties

See ISO 4665/1.

9.2 Determination of resistance to cracking

See ISO 1431/1.

9.3 Climatic conditions

9.3.1 Classes of climate

Climates are divided into five main classes, each subdivided into several types.

Annex C gives details of two such classifications in use throughout the world.

The classification of climates given is such that significant differences are to be expected between the effects of exposure of rubbers to each of the climatic conditions.

9.3.2 Climatic observations

The general description of the climate at the site by class, type and special indications shall, when necessary, be supplemented by the following detailed observations.

9.3.2.1 Temperature

a) monthly mean of daily maxima;

b) monthly mean of daily minima;

c) monthly maximum and minimum.

9.3.2.2 Relative humidity

a) monthly mean of daily maxima;

b) monthly mean of daily minima;

c) monthly range.

9.3.2.3 Radiation (if not dealt with under clause 7)

Monthly total hours of sunshine.

9.3.2.4 Precipitation

Monthly total amount.

9.3.2.5 Other observations

Other observations such as wind strength and direction, incidence and nature of any atmospheric pollution, for example ozone, total hours of dew and rain, total incident energy and any special local features may also be recorded.

10 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) exposure aspect,

2) location and details of exposure site,

3) class and type of climate, quoting reference authority (see annex C),

4) whether test pieces were strained or unstrained — in the case of strained test piece, the elongation(s) and procedure used,

5) nature of backing, support and attachments, if used,

6) whether a glass lid was used,

7) procedure for determining exposure stages, including method used to determine radiation dosage, if used,

8) details of washing, if any;

d) test results:

1) exposure stages used,

2) climatological data,

3) resistance to cracking in case of strained test pieces,

4) presentation of results as required by ISO 4665/1;

e) the date of test.

Annex A

Use of blue dyed wool standards to measure light dosage

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

A.1 General

The blue dyed wool standards were developed for textile testing and historically have been used with rubber because of their availability. Because, in general, there is a need to expose rubber for longer periods than those normally used for testing the light fastness of textiles, the consecutive use of the number 7 standard has been introduced.

Owing to the differences between the spectral sensitivity of the blue wool standards and the rubber materials, there is considerable doubt about the blue wool standards for this application. However, their ready availability and the fund of data based on their use ensures that there is still a demand for their application in exposure tests on rubber.

A.2 Procedure

Expose a set of ISO blue dyed wool standards comprising one strip each from numbers 1 to 7 simultaneously.

Use the standards to determine the stages of radiation dosage (exposure stages) in accordance with the table by comparing the difference in colour between the exposed and unexposed blue standards with the contrast 4 on the grey scale; thus, stage 1/1 is reached when standard 1 gives a contrast equal to 4 on the grey scale; 2/1 when standard 2 shows similar contrast, and in the same manner to stage 7/1 showing a contrast of 4 on the grey scale.

NOTE — The duration of stage 7/1 is about 1 year in natural daylight in temperate climates.

Inspect the blue standards as frequently as necessary to determine when each exposure stage is reached.

At stage 7/1, discard the blue standards, mount a second fresh standard 7 and continue exposure until this second standard 7 shows a contrast with the unexposed standard 7 equal to 4 on the grey scale. This stage is designated 7/2.

Then discard the second standard 7 and mount a third fresh standard 7. Stage 7/3 is reached when this standard in turn gives a contrast of 4.

Repeat this procedure as often as required, giving stages 7/4,..., 7/n.

NOTE — Consecutive exposure of No. 7 standard should only be employed when no better alternative is available.

Exposure stages

Stage	Description
1/1	Blue standard 1 to grey scale contrast 4
2/1	Blue standard 2 to grey scale contrast 4
3/1	Blue standard 3 to grey scale contrast 4
4/1	Blue standard 4 to grey scale contrast 4
5/1	Blue standard 5 to grey scale contrast 4
6/1	Blue standard 6 to grey scale contrast 4
7/1	First blue standard 7 to grey scale contrast 4
7/2	Second blue standard 7 to grey scale contrast 4
7/n	n^{th} blue standard 7 to grey scale contrast 4

Annex B

Suppliers of blue dyed wool standards and grey scale

(This annex does not form an integral part of the Standard.)

B.1 Suppliers of blue dyed wool standards

Sets of the ISO dyed wool light fastness standards can be obtained from the following organizations :

British Standards Institution
3 York Street
Manchester M2 2AT
United Kingdom

Beuth-Vertrieb GmbH
Burggrafenstrasse 4-7
D-1000 Berlin 30
Germany, F.R.

American Society for Testing and Materials
1916 Race Street
Philadelphia
Pennsylvania 19103
USA

Eidgenössische Materialprüfungs-und Versuchsanstalt
Unterstrasse 11
CH-9000 St Gallen
Switzerland

Association pour la détermination de la solidité
des teintures et impressions sur textiles
12, rue d'Anjou
F-75008 Paris
France

Japanese Standards Association
1-24 Akasaka 4
Minato-ku
Tokyo
Japan

and other countries.

B.2 Suppliers of grey scale

The grey scale for assessing change in colour can be obtained from the following organizations:

British Standards Institution
3 York Street
Manchester M2 2AT
United Kingdom

Society of Dyers and Colourists
PO Box 244, Perkin House
82 Gratton Road
Bradford BD1 2JB
West Yorks
United Kingdom

Beuth-Vertrieb GmbH
Burggrafenstrasse 4-7
D-1000 Berlin 30
Germany, F.R.

Association pour la détermination de la solidité
des teintures et impressions sur textiles
12, rue d'Anjou
F-75008 Paris
France

Eidgenössische Materialprüfungs-und Versuchsanstalt
Unterstrasse 11
CH-9000 St Gallen
Switzerland

Japanese Standards Association
1-24 Akasaka 4
Minato-ku
Tokyo
Japan

American Association of Textile Chemists and Colorists
PO Box 12215
Research Triangle Park
North Carolina 27709
USA