INTERNATIONAL STANDARD

ISO 3865

Fifth edition
2020-01

Rubber, vulcanized or thermoplastic — Methods of test for staining in contact with organic material

Caoutchouc vulcanisé ou thermoplastique — Méthodes d'essai pour déterminer le tachage lors du contact avec des matières organiques

Reference number
ISO 3865:2020(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fifth edition cancels and replaces the fourth edition (ISO 3865:2005), which has been technically revised. The main changes to the previous edition are as follows:

— the list of normative references has been updated in Clause 2.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

When rubber is in contact with organic material, such as paints, varnishes, plastics or rubber, under conditions of heat, pressure and light, staining can occur on the surface in contact with the rubber, on the surface adjacent to the rubber or on the surface of the organic material which covers the rubber. In addition, in the presence of water, constituents of the rubber can be leached out, which can cause staining on surfaces with which the water subsequently comes into contact.

Rubber, vulcanized or thermoplastic — Methods of test for staining in contact with organic material

WARNING 1 — Persons using this document shall be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of any other restrictions.

WARNING 2 — Certain procedures specified in this document might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This document specifies three methods for estimating the staining of organic finishes (subsequently referred to as "organic material") by vulcanized or thermoplastic rubber, as defined in Clause 3:

— method A: contact staining and migration staining;

— method B: extraction staining;

— method C: penetration staining;

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 105-A01, *Textiles — Tests for colour fastness — Part A01: General principles of testing*

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

ISO 105-B01, *Textiles — Tests for colour fastness — Part B01: Colour fastness to light: Daylight*

ISO 188, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests*

ISO 1382, *Rubber — Vocabulary*

ISO 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures*

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*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1382 and the following 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/

3.1
contact staining
stain which occurs on the surface of the organic material directly in contact with the rubber

3.2
migration staining
stain which occurs on the surface of the organic material surrounding the contact area

3.3
extraction staining
stain which occurs on the surface of the organic material as a result of contact with a liquid containing leached out constituents of the rubber

3.4
penetration staining
stain which occurs on the surface of a veneer layer of an organic material which is bonded to the rubber surface

4 Principle

4.1 Method A — Determination of contact straining and migration staining

The rubber to be tested is placed in direct contact with the specified organic material and then exposed to heat and/or artificial light.

4.2 Method B — Determination of extraction staining

The rubber to be tested is subjected to a test liquid which afterwards contacts the organic material. This may be followed by irradiation of the organic material.

4.3 Method C — Determination of penetration staining

A light-coloured veneer or lacquer of specified material is applied to the rubber to be tested and then exposed to heat followed by artificial light.

4.4 Assessment of staining

The stain is assessed qualitatively by visual inspection or quantitatively by comparison with a grey scale or by using a reflectance spectrometer.

5 Apparatus

Use the following apparatus for the methods indicated.

5.1 Ageing air oven, in accordance with ISO 188.

5.2 Artificial light source, consisting of a xenon-arc lamp, filtered to give a spectral distribution corresponding to that of sunlight, as specified in ISO 4892-2 and in 9.1 and 9.2.

5.3 Irradiation chamber, containing the lamp and the test piece racks, designed to meet the requirements in 9.3 and 9.4.

5.4 Thermocouple or **black panel thermometer**, as specified in ISO 4892-1 for measurement of surface temperature.

5.5 Suitable apparatus for measuring the light intensity over the range of wavelengths given in 9.1 (recommended, although not mandatory).

5.6 Blue dyed wool standards, as specified in ISO 105-B01.

5.7 Grey scale, as specified in ISO 105-A02.

5.8 Reflectance spectrometer, operating in the range 400 nm to 600 nm.

5.9 Beaker or **dripping apparatus** (in method B).

5.10 Dripping and drying frame, for finishing with lacquer (in method C).

6 Test pieces

6.1 Rubber test pieces

Rubber test pieces shall be rectangular in shape, of uniform thickness and preferably cut from sheet 2 mm ± 0,2 mm thick. The minimum dimensions shall be:

— for method A: 12 mm × 25 mm;

— for method B.1: 25 mm × 150 mm:

— for method B.2: 3 pieces with a total mass of 5 g ± 0,2 g;

— for method C: 12 mm × 25 mm.

Test pieces for method C shall be cut from samples prepared in accordance with 6.3.

Test pieces may also be cut from finished products, in which case they may be cleaned of extraneous contamination before test with a 2 % non-alkaline soap solution.

6.2 Metal or plastics panels for methods A and B

The dimensions of metal or plastics panels shall meet the requirements specified in 8.1 or 8.2, but otherwise are not critical.

Panels shall be coated with a material to be agreed between purchaser and supplier. Unless otherwise specified, a white acrylic-based stoving enamel shall be used. This lacquer shall be dried in the oven (5.1) for 30 min at 125 °C and tests shall commence between 24 h and 48 h after drying. If other times are used, these shall be stated in the test report.

6.3 Test piece preparation for method C

6.3.1 General

A white or light-coloured non-discolouring rubber veneer, of a composition to be agreed between purchaser and supplier, shall be applied under pressure to a sheet of the test rubber. The veneer shall either be vulcanized with the test rubber or be applied as a paint on a previously prepared sheet of vulcanized or thermoplastic rubber.

As agreed between the interested parties, test pieces may be taken from finished products with light-coloured veneered or lacquered surfaces, such as white tyre sidewalls. The method of construction and the sample thickness shall be mentioned in the test report.

6.3.2 Vulcanized test pieces and veneers

All mixing shall be carried out in a thoroughly cleaned mixer, preferably in accordance with ISO 2393. The rubber shall be sheeted out to a thickness of 2,0 mm ± 0,2 mm, and protected on both sides by an inert material, such as starched cambric fabric or polyethylene sheet, until tested. A test piece shall be cut out to the required mould dimensions.

The rubber veneer shall be calendered to a thickness of 0,5 mm ± 0,05 mm and stiffened on at least one side with a protective aluminium sheet.

At the time of coating, one protective layer shall be removed from both the test rubber and the veneer, and the two exposed surfaces pressed firmly together, ensuring that the aluminium sheet remains on the external side of the rubber veneer. Pressure may be applied by a platen press or by rollers.

The composite body, including the aluminium sheet, shall be moulded and vulcanized in a platen press, taking care that the veneer and aluminium sheet are situated at the bottom side of the mould. The conditions of vulcanization shall be included in the test report. The protective surface on the veneer shall be left until used.

6.3.3 Lacquered test pieces

Test pieces shall be immersed in a white, non-staining lacquer at a depth of 25 mm. The test pieces shall be hung on a suitable rack and dried. After drying, they shall be immersed a second time in the lacquer, then dried until the surface is non-tacky.

Instead of lacquer, a paste prepared of the unvulcanized veneer in a suitable solvent (volume ratio 1:6) may be used. The procedure shall be the same as for lacquer.

Aluminium foil dipped in the lacquer/paste may be used as a blank.

The thickness of the lacquer and paste layer shall be agreed between the interested parties. If not otherwise specified, the thickness of the lacquer layer shall be about 0,1 mm, and the thickness of the paste layer shall be about 0,16 mm.

6.4 Blanks and reference samples

6.4.1 Blanks

Blanks shall be prepared and treated in the same manner as the samples to be tested, except that the rubber to be tested shall be replaced by an inert material. A suitable inert material, such as an aluminium sheet about 0,4 mm to 0,6 mm thick, may be used as an alternative to the rubber slab.

6.4.2 Reference samples

Reference samples shall be prepared in the same way and with the same construction as the samples to be tested (6.1 to 6.3), but shall be protected from irradiation in an appropriate manner, i.e. by an appropriate covering during the irradiation exposure period. Also, part of the sample can be covered to act as a reference sample.

6.5 Conditioning of samples and test pieces

For all test purposes, the minimum time between vulcanization and testing shall be 16 h. For non-product tests, the maximum time between vulcanization and testing shall be 4 weeks.

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

7 Number of test pieces

At least two test pieces shall be used.

8 Procedure

8.1 Method A — Contact staining and migration staining

A test piece in accordance with 6.1, method A, shall be used.

Place the test piece between two painted metal or plastics panels (see 6.2). The dimensions of the panels shall be such that a rim of at least 20 mm width around the test piece is left uncovered. If two or more test pieces are placed between the same panels, the distance between the test pieces shall be at least 40 mm.

Apply a pressure of 7 kPa ± 1 kPa, calculated on the area of the test piece, to the assembly. Store the loaded assembly in the oven (5.1) at 70 °C ± 2 °C for 24 $^{0}_{-2}$ h, taking care that no other volatile or vapour-producing materials that might affect staining are in the oven. After removal from the oven, wash one panel with distilled water containing about 2 % of a detergent which is free from alkalinity, and examine for both contact staining and migration staining in accordance with Clause 10.

Expose the second panel, without the rubber test piece, to artificial light, the recommended conditions of irradiation being as given in Clause 9. Then wash the panel with distilled water containing about 2 % of a detergent which is free from alkalinity and examine for both contact staining and migration staining in accordance with Clause 10.

Test a blank assembly, in which the rubber is replaced by aluminium, at the same time and assess the degree of staining relative to the blank. Expose no panel more than once.

If the action of heat only is required, the irradiation part of the procedure can be omitted.

8.2 Method B — Extraction staining

8.2.1 General

A test piece in accordance with 6.1, method B.1 or B.2, shall be used. The test liquid shall be distilled water or diluted ethanol.

The test liquid is first brought into contact with the rubber test piece and then into contact with the painted test panel as described below for method B.1 or method B.2.

After this treatment, wash the panel with distilled water containing about 2 % of a detergent which is free from alkalinity, and examine for staining in accordance with Clause 10.

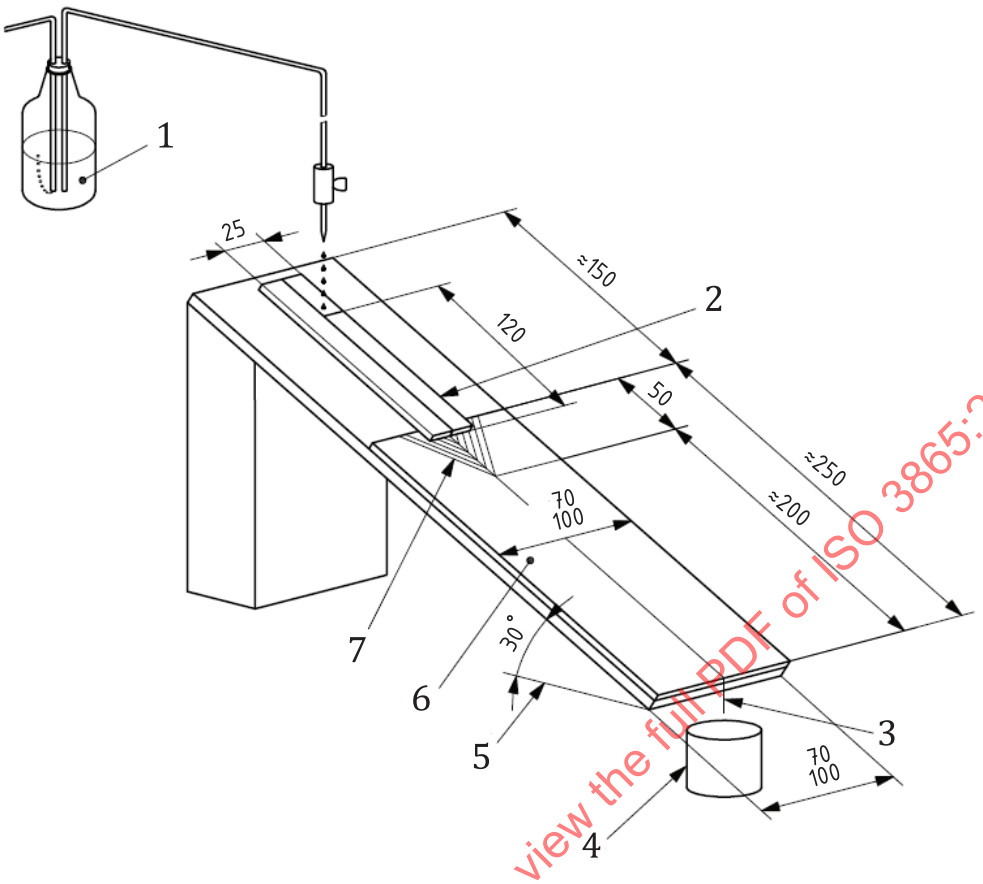
If required, the panel may then be exposed to artificial light, the recommended conditions of irradiation being as given in Clause 9. Then wash the panel with distilled water containing about 2 % of a detergent which is free from alkalinity and examine the panel in accordance with Clause 10.

Test a blank assembly at the same time and assess the degree of staining relative to the blank.

8.2.2 Method B.1

Drip the test liquid onto the test piece, at a rate of 1 dm^3 in 24 h, as shown in Figure 1. The liquid runs along the test piece and subsequently along a cotton thread placed on the painted metal or plastics panel. Continue the dripping for 24 $^{0}_{-2}$ h.

Dimensions in millimetres



Key

1	test liquid (dripping rate 1 dm^3/24 h)	5	horizontal
2	rubber test piece (25 mm × 150 mm)	6	painted metal or plastics panel
3	thread	7	segment of filter paper
4	drip container		

Figure 1 — Dripping apparatus for method B.1

8.2.3 Method B.2

Immerse three rubber pieces with a total mass of 5 g ± 0,2 g in a beaker in 50 ml ± 1 ml of the test liquid at standard laboratory temperature. After 24 $^{0}_{-2}$ h, replace the rubber with test panels, according to 6.2, for another period of 24 $^{0}_{-2}$ h.

8.3 Method C — Penetration staining

Use a test piece in accordance with 6.3.

Place the test piece in the oven (5.1) for 24 $^{0}_{-2}$ h at 70 °C ± 2 °C unless otherwise specified, taking care that the oven contains no volatile or vapour-producing substances that might affect staining. Remove the protective aluminium sheet, then expose the coated surface of the test piece to artificial light, the recommended conditions of irradiation being as given in Clause 9. Then wash the panel with distilled water containing about 2 % of a detergent which is free from alkalinity and examine for staining in accordance with Clause 10.

Test a blank assembly at the same time and assess the degree of staining relative to the blank.

9 Recommended conditions of irradiation

9.1 Intensity

The light source should be a xenon-arc lamp (5.2) which gives an irradiance (radiant flux density) specified in ISO 4892-2 at the test piece surface.

9.2 Irradiation time

Unless otherwise specified, the preferred irradiation period should be 24 h, 48 h or 168 h.

Alternatively, test pieces may be irradiated together with the blue dyed wool standards (5.6) until one of the standards, 3, 4 or 6, chosen in advance, shows between exposed and unexposed areas a contrast equal to grade 4 of the grey scale (5.7).

9.3 Surface temperature

The surface temperature in the plane of the test piece should be 55 °C ± 3 °C when measured with a black panel thermometer (5.4).

9.4 Local distribution of test pieces

When several test pieces are exposed to irradiation at the same time, care should be taken that all test pieces are irradiated equally. The intensity of irradiation should not vary by more than 10 % from the mean at any point of the irradiated surface.

This condition is best achieved by allowing the test pieces to rotate about the lamp.

10 Evaluation of degree of staining

10.1 General

Assess the severity of staining in accordance with one of the following methods: 10.2; 10.3; 10.4; or Table 1.

10.2 Qualitative assessment

Make a visual assessment of the degree of staining relative to a blank or a reference sample (6.4).

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

a) hue:
   1) more blue or less blue;
   2) more green or less green;
   3) more red or less red;
   4) more yellow or less yellow.

b) purity:
   1) duller;

2) brighter.

c) lightness:

1) lighter;

2) darker.

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

10.3 Assessment using a grey scale

Carry out a visual assessment of the colour changes, following the principles established in ISO 105-A01, by comparing the contrasts existing between the exposed test piece and the blank or reference sample with the rating on the grey scale, as specified in ISO 105-A02. The rating of colour change is the grade on the grey scale which shows an equivalent contrast to that existing between the exposed test piece and the blank or reference sample.

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

10.4 Assessment using a reflectance spectrometer

10.4.1 If a quantitative measurement (other than that obtainable by the use of the grey scale) of colour change is required, reflectance measurements shall be made with the reflectance spectrometer (5.8) operating in the range 400 nm to 600 nm. Measurements shall be made relative to a blank or reference sample at a minimum of three wavelengths (for example, 445 nm, 555 nm and 600 nm). In each case the reflectance spectrometer shall be calibrated using barium sulfate ($BaSO_4$).

If $\rho(\lambda)$, $\rho(\lambda)_B$, and $\rho(\lambda)_0$ are, respectively, the reflectance readings at wavelength λ for the test panel, the blank panel and the reference panel, the measure of colour change is taken as the following difference:

$\rho(\lambda) - \rho(\lambda)_B = \Delta\,\rho(\lambda)_B$ or $\rho(\lambda) - \rho(\lambda)_0 = \Delta\,\rho(\lambda)_0$

The difference is given in per cent, relative to the white medium. Negative values indicate darkening and positive values lightening.

Table 1 — Staining graduation

Qualitative assessment (see 10.2)	**Grey scale assessment** (see 10.3)		**Reflectance difference $\Delta\rho(\lambda)$ assessment** % (see 10.4.1)	
	White coating	**Coloured coating**	**White coating**	**Coloured coating**
No staining	5 to 4	5	0 to 4	0 to 2
Slight staining	3 to 2	4 to 3	> 4 to 10	> 2 to 5
Moderate staining	1	2	> 10 to 25	> 5 to 12
Severe staining	< 1	1	> 25	> 12

10.4.2 Calculate the colour difference (total difference ΔE^*_{ab} between two colours) and the component of colour difference [lightness difference (ΔL^*_{ab}), chroma difference (ΔC^*_{ab}) and hue difference (ΔH^*_{ab})] (see ISO/CIE 11664-4).