INTERNATIONAL STANDARD

ISO 3865

Third edition
1997-12-01

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 les matières organiques

Reference number
ISO 3865:1997(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 3865 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests.*

This third edition cancels and replaces the second edition (ISO 3865:1983), which has been technically revised. The main changes are the addition of a principle clause and a new method (method B2) and the deletion of the mercury arc lamp.

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Printed in Switzerland

Introduction

When rubber is in contact with organic material, such as paints or varnishes, plastics, rubber, under conditions of heat, pressure and light, staining may 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 may be leached out which may 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 - Persons using this International Standard shall be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, asssociated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies three methods for estimating the staining of organic finishes (in the following called "organic material") by vulcanized or thermoplastic rubber:

method A: contact and migration stain;

method B: extraction stain;

method C: penetration stain;

as defined in clause 3.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated are valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 105, *Textiles - Tests for colour fastness*

part A01:1994, *General principles of testing*

part A02:1993, *Gray scale for assessing change in colour*

part B01:1994, *Colour fastness to light: Daylight*

ISO 188:1982, *Rubber, vulcanized - Accelerated ageing or heat resistance tests*

ISO 2393:1994, *Rubber test mixes - Preparation, mixing and vulcanization - Equipment and procedures*

ISO 4665-3:1987, *Rubber, vulcanized - Resistance to weathering - Part 3: Methods of exposure to artificial light*

3 Definitions

For the purpose of this International Standard, the following definitions apply:

3.1 contact stain: The stain which occurs on the surface of the organic material directly in contact with the rubber;

3.2 migration stain: The stain which occurs on the surface of the organic material surrounding the contact area;

3.3 extraction stain: The 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 stain: The 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 and migration stain)

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 stain)

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

4.3 Method C (Determination of penetration stain)

A light-coloured veneer or lacquer of specified material is applied to the rubber to be tested. The veneer is subjected to irradiation with a foregoing heating.

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

The following apparatus is used for the methods indicated:

5.1 Ageing air oven conforming to ISO 188.

5.2 Artificial light source consisting of a xenon arc lamp, filtered to give a spectral distribution corresponding to that of sunlight, conforming to ISO 4665-3 and fulfilling the requirements given in 9.1; methods A, B and C.

5.3 Irradiation chamber, containing the lamp and the test piece racks, and designed to meet the requirements specified in 9.3 and 9.4; methods A, B and C.

5.4 Thermocouple or **black panel thermometer** (see ISO 4665-3) for measurement of surface temperature; methods A, B and C.

5.5 Suitable apparatus for measuring the light intensity over the range of wavelengths given in 9.1; methods A, B and C. (Recommended, although not mandatory.)

5.6 Blue dyed wool standards as specified in ISO 105-B01; methods A, B and C.

5.7 Grey scale as specified in ISO 105-A02; methods A, B and C.

5.8 Reflectance spectrometer operating in the range 400 nm to 600 nm; methods A, B and C.

5.9 Beaker or **dripping apparatus**; method B.

5.10 Dripping and drying frame for finishing with lacquer; 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 x 25 mm
for method B1 25 mm x 150 mm
for method B2 3 pieces with a total mass of 5 g ±0,2 g
for method C 12 mm x 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. If not 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 be commenced between 24 h and 48 h after drying.

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. Sheet out the rubber to a thickness of 2,0 mm ±0,2 mm, protect it on both sides by an inert material, such as starched cambric fabric or polyethylene sheet, until tested, and cut out a test piece to the required mould dimensions.

Calender the rubber veneer to a thickness of 0,5 mm ±0,05 mm and stiffen it on at least one side with a protective aluminium sheet.

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

Mould and vulcanize the composite body, including the aluminium sheet, 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. Leave the protective surface on the veneer until use.

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) can be used. The procedure shall be the same as for lacquer.

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

The thickness of the lacquer layer shall be 0,1 mm ±0,02 mm and of the paste layer 0,16 mm ±0,04 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 would be an aluminium sheet about 0,4 to 0,6 mm thick, to be used instead of the rubber slab.

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

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 h +0/-2 h, taking care that no other volatile or vapour-producing materials that might affect stain 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 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 and migration staining in accordance with clause 10.

Test a blank assembly (the rubber is replaced by alumina) at the same time and assess the degree of staining relative to the blank. Expose no panel more than once.

8.1.1 Method A1
If the action of heat only is required, the irradiation part of the procedure may be omitted.

8.1.2 Method A2
If the action of light only is required, the exposure-to-heat part of the procedure may be omitted. In this case, fasten the test piece to one panel with a metal clamp, observing the requirements for panel dimensions and pressure given in 8.1, and irradiate this assembly. Then examine the area of the panel around the edge of the test piece for migration staining.

8.2 Method B - Extraction stain

A test piece in accordance with 6.1, method B1 or B2, shall be used.

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 B1 or Method B2.

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.

8.2.1 Method B1
Drip the test liquid onto the test piece, at a rate of 1 dm^3 in 24 h, as shown in the figure. 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 h +0/-2h.

8.2.2 Method B2
3 rubber pieces with a total mass of 5 g ±0,2 g are immersed in a beaker in 50 ml ±1 ml of the test liquid at room temperature. After 24 h +0/-2 h the rubber is replaced by test panels according to 6.2 for another period of 24 h +0/-2 h.

8.3 Method C - Penetration stain

A test piece in accordance with 6.3 shall be used.

Place the test piece in the oven (5.1) for 24 h +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 stain. Remove the protective aluminium foil, 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 is a xenon arc lamp (5.2) which gives an irradiance (radiant flux density) at the test piece surface of 1000 W/m^2 ±200 W/m^2 in the wavelength range 300 nm to 830 nm.

9.2 Irradiation time

Unless otherwise specified, the preferred irradiation period shall 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 shall 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 shall be taken that all test pieces are irradiated equally. The intensity of irradiation should not vary by more tyhan 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

Assess the severity of staining in accordance with one of the following methods 10.1, 10.2, 10.3 and the table.

10.1 Qualitative assessment

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

10.2 Assessment using a grey scale

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

10.3 Assessment using a reflectance spectrometer

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 difference:

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

The difference is given in percent, related to the white medium. Negative values indicate darkening and positive values lightening.

Table - Staining graduation

Assessment according to				
10.1 (qualitative)	10.2 (grey scale)		10.3 (reflectance difference $\Delta\,\rho(\lambda)$ in %)	
	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	10 to 12
Severe staining	<1	1	>25	>12

11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) sample details:

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

2) compound details and cure conditions, if known;

3) dimensions, specifications and method of preparation of test pieces, organic material, rubber veneer and lacquer as appropriate;

c) test details:

1) a reference to the method used [A (A1 or A2), B (B1 or B2) or C];

2) test liquid used (for method B);

3) details of the irradiation source, and its distance from the test piece;

4) the time and temperature of irradiation;

5) any deviations from the standard procedure;

Dimensions in millimetres

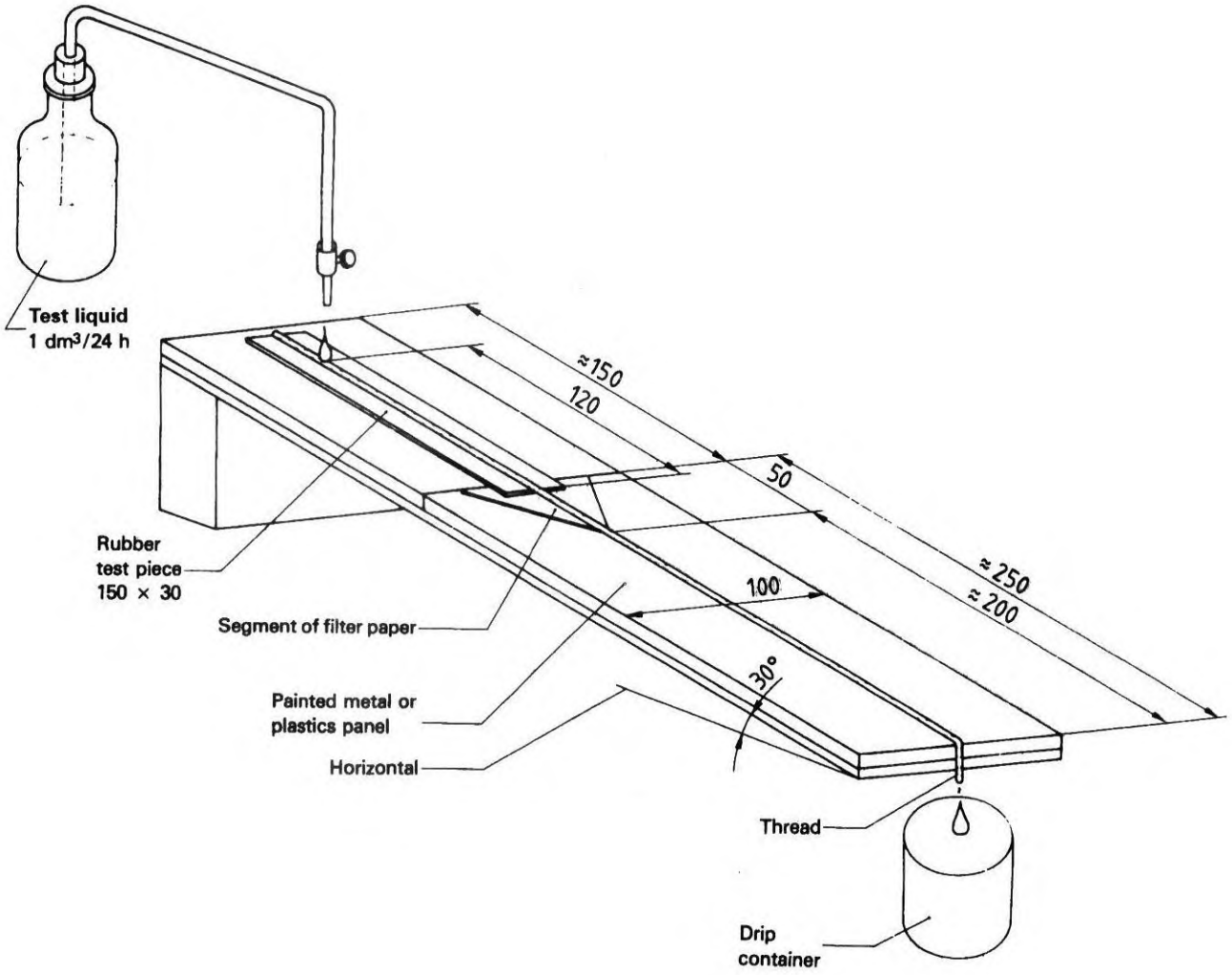

Figure 1 — Dripping apparatus for method B1

d) test results:

1) number of test pieces used;

2) the method of assessment of degree of staining

3) the individual test results according to clause 10, which may be expressed in one of the following ways
- qualitative description of the staining (10.1);
- the gray scale (10.2);
- change in reflectance (10.3);

4) occurence of migration staining with methods A, A1 and A2:

e) the date of test.