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

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

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FOREWORD

ISO (the International Organization for Standardization), is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

International Standard ISO 3865 was drawn up by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the Member Bodies in April 1975.

It has been approved by the Member Bodies of the following countries :

Australia	Italy	Switzerland
Belgium	Mexico	Thailand
Brazil	New Zealand	Turkey
Canada	Poland	United Kingdom
Czechoslovakia	Romania	U.S.A.
France	South Africa, Rep. of	U.S.S.R.
Germany	Spain	Yugoslavia
Hungary	Sweden	

No Member Body expressed disapproval of the document.

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

1 SCOPE AND FIELD OF APPLICATION

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

method A : contact and migration stain;

method B : extraction stain;

method C : penetration stain.

2 REFERENCES

ISO/R 105/I, *Tests for colour fastness of textiles — First series.*

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

ISO/R 879, *Plastics — Determination of resistance of plastics to colour change upon exposure to light of a xenon lamp.*

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

3 DEFINITIONS

When vulcanized rubber is in contact with organic material, such as paints and plastics or 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 of surfaces with which the water subsequently comes into contact.

3.1 contact stain : The stain which occurs on the surface directly in contact with the rubber.

3.2 migration stain : The stain which occurs on the surface surrounding the contact area.

3.3 extraction stain : The stain caused by contact with water containing leached-out constituents of the rubber.

3.4 penetration stain : The staining of a veneer layer of an organic material bonded to the rubber surface.

4 APPARATUS

The following apparatus is used for the methods indicated :

4.1 Air circulating oven conforming to ISO 188; methods A and C.

4.2 Xenon arc lamp, filtered to give a spectral distribution corresponding to that of sunlight, conforming to ISO/R 879; methods A, B and C.

4.3 Mercury arc lamp, with filter for absorption of radiation below 280 nm (alternative to xenon lamp); methods A, B and C.

4.4 Thermocouple or **black panel thermometer,** for measurement of surface temperature; methods A, B and C.

4.5 Suitable apparatus for measuring the light intensity over the range of wavelengths given in 8.1; methods A, B and C. (Recommended, although not mandatory.)

4.6 Blue dyed wool standards as specified in ISO/R 105/I, part 11, sub-clause 3.1.1; methods A, B and C.

4.7 Grey scale as specified in ISO/R 105/I, part 2; methods A, B and C.

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

4.9 Beaker and **dripping apparatus;** method B.

4.10 Dripping and drying frame; method C.

5 TEST PIECES

5.1 Rubber test pieces

Rubber test pieces shall be rectangular in shape, of uniform thickness and preferably cut from sheet 2 ± 0,2 mm thick. The minimum dimensions shall be 25 mm × 12 mm for method A and 150 mm × 25 mm for method B. The test piece for method C shall have minimum dimensions of 25 mm × 12 mm and shall be cut from samples prepared in accordance with 5.3.

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.

5.2 Metal or plastics panels for methods A and B

The dimensions of painted metal or plastics panels to be tested for staining caused by the rubber test pieces are not critical, but the requirements given in the test procedure must be complied with.

Panels shall be coated with a lacquer 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 air circulating oven (4.1) for 30 min at 125 °C and tests shall be commenced between 24 and 48 h after drying.

5.3 Test piece preparation for method C

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, and the composite shall be vulcanized.

All mixing shall be carried out on a thoroughly cleaned mill, preferably in accordance with ISO 2393. Sheet out the rubber to a thickness of 2,0 ± 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 ± 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 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.

NOTES

1 Alternatively, test pieces vulcanized according to 5.3 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.

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

The thickness of the lacquer layer shall be 0,1 ± 0,02 mm.

2 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.

5.4 Blanks and reference samples

5.4.1 *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.

5.4.2 *Reference samples*

Reference samples differ from blanks in so far as they are prepared in the same way and with the same construction as the samples to be tested (5.1 to 5.3) but are protected from irradiation in an appropriate manner, i.e. by an appropriate covering during the irradiation exposure period.

5.5 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.

6 NUMBER OF TEST PIECES

Normally one test piece will be sufficient.

7 PROCEDURE

7.1 Method A — Contact and migration stain

A test piece in accordance with 5.1, method A, shall be used.

Place the test piece between two painted metal or plastics panels (see 5.2). The dimensions of the panels shall be such that a rim of at least 20 mm width around the test piece is left uncovered. Apply a pressure of 7 ± 1 kPa, calculated on the area of the test piece, to the assembly. If two or more test pieces are placed between the same panels, the distance between the test pieces shall be at least 40 mm. Store the loaded assembly in the air circulating oven (4.1) at 70 ± 2 °C for 24 $^{0}_{-2}$ 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 9.

Expose the second panel, without the rubber test piece, to artificial light, the recommended conditions of irradiation being as given in clause 8. 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 9.

Test a blank assembly at the same time and assess the degree of staining relative to the blank. Expose no panel more than once.

7.1.1 *Method A 1*

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

7.1.2 *Method A 2*

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 given in 7.1, and irradiate this assembly. Then examine the area of the panel around the edge of the test piece for migration staining.

7.2 Method B — Extraction stain

A test piece in accordance with 5.1, method B, shall be used.

Drip distilled water onto the test piece, at a rate of 1 l in 24 h, as shown in the figure. The water 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.

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 9.

If required, the panel may then be exposed to artificial light, the recommended conditions of irradiation being as given in clause 8. 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 9.

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

7.3 Method C — Penetration stain

A test piece in accordance with 5.3 shall be used.

Expose the coated surface of the test piece to artificial light, the recommended conditions of irradiation being as given in clause 8. Then wash the test piece with distilled water containing about 2 % of a detergent which is free from alkalinity, and examine for staining in accordance with clause 9.

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

Dimensions in millimetres

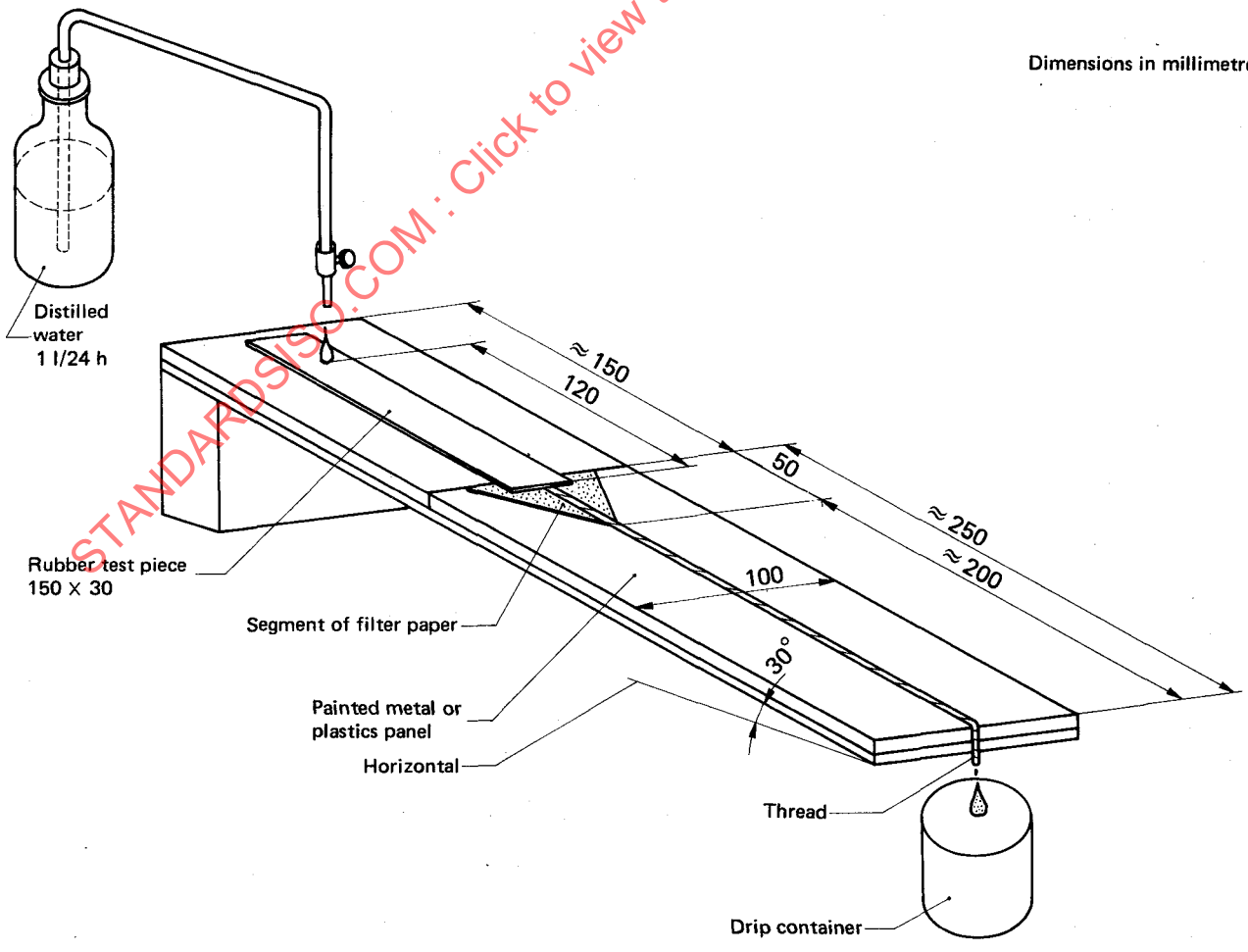

FIGURE — **Dripping apparatus**

8 RECOMMENDED CONDITIONS OF IRRADIATION

8.1 Intensity

The preferred light source is a xenon arc lamp (4.2) which gives an irradiation intensity at the test piece surface of $1\,000 \pm 200$ W/m² in the wavelength range 300 to 830 nm. This intensity is conveniently achieved with lamps of nominal power rating 1 500 W. Lamps of higher power (for example 6 000 W) may be used but these will give a higher irradiation intensity.

An alternative light source is a mercury arc lamp (4.3) which gives an irradiation intensity at the test piece surface of 15 to 45 W/m² in the wavelength range 280 to 400 nm. This intensity is conveniently achieved with lamps of nominal power rating 500 W.

8.2 Irradiation time

Unless otherwise specified, the preferred irradiation period shall be as follows :

xenon arc lamp (1 500 W)	24,	48,	150 h
mercury arc lamp	2,	4,	8 h

Alternatively, with a xenon arc lamp only, test pieces may be irradiated together with the blue dyed wool standards (4.6) 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 (4.7).

8.3 Surface temperature

The surface temperature in the plane of the test piece shall be 60 ± 15 °C when measured with a black panel thermometer (4.4).

8.4 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 than 10 % from the mean at any point on the irradiated surface.

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

When using a mercury arc lamp, the test pieces should not be placed centrally below the lamp but should be arranged in an annular zone between 90 and 300 mm diameter upon a turntable rotating at approximately 3 rev/min.

9 EVALUATION OF DEGREE OF STAINING

Assess the severity of staining in accordance with one of the following methods 9.1, 9.2, 9.3 and the table.

9.1 Visual assessment

Make a visual assessment of the degree of staining relative to a blank or a reference sample (5.4).

9.2 Assessment using a grey scale

It may be advantageous if the degree of colour difference is judged with the aid of the grey scale (4.7).

9.3 Assessment using a reflectance spectrophotometer

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 spectrophotometer (4.8) operating in the range 400 to 600 nm. Measurements shall be made relative to a blank or reference sample at a minimum of three wavelengths (for example 445, 555 and 600 nm). In each case the reflectance spectrophotometer shall be calibrated using barium sulphate ($BaSO_4$).

If R and R_0 are respectively the reflectance readings for the sample panel and the blank or reference panel, the measure of colour change is taken as $R - R_0$.

Hence, negative values indicate darkening and positive values lightening.

TABLE – **Staining graduation**

Assessment according to		
9.1 (visual)	**9.2** (grey scale)	**9.3** [reflectance difference $(R - R_0)$]
No staining	5 to 4	0 to 4
Slight staining	3 to 2	> 4 to 10
Moderate staining	1	> 10 to 25
Severe staining	< 1	> 25

10 TEST REPORT

The test report shall include the following particulars :

a) the test piece dimensions;

b) reference to this International Standard;

c) the method used [A, (A 1, A 2), B or C];

d) the dimensions and nature of the organic material;

e) time and temperature of storage;

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

g) the time and temperature of irradiation;

h) the method of assessment of degree of staining;

i) the individual test results according to clause 9, which may be expressed in one of the following ways :

qualitative description of the staining (9.1); the grey scale (9.2); change in reflectance (9.3);

j) conditions of vulcanization for coated rubber test pieces;

k) specifications of the rubber veneer or lacquer if applicable;

l) occurrence of migration staining with methods A, A 1 and A 2.