Paints and varnishes — Determination of hiding power —

Part 3: Determination of contrast ratio of light-coloured paints at a fixed spreading rate

1 Scope

This part of ISO 6504 describes methods for determining the opacity (by contrast ratio measurement) given by paint films of white or light colours of tristimulus value Y greater than 25, applied at a spreading rate of 20 m^2/l to a black and white chart or to colourless transparent polyester foil. In the latter case, the tristimulus value Y is measured subsequently over black and white glass panels.

2 Normative references

The following referenced documents are indispensable for the application 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 1513, *Paints and varnishes — Examination and preparation of samples for testing*

ISO 2811-1, *Paints and varnishes — Determination of density — Part 1: Pyknometer method*

ISO 2811-2, *Paints and varnishes — Determination of density — Part 2: Immersed body (plummet) method*

ISO 2811-3, *Paints and varnishes — Determination of density — Part 3: Oscillation method*

ISO 2811-4, *Paints and varnishes — Determination of density — Part 4: Pressure cup method*

ISO 3251, *Paints, varnishes and plastics — Determination of non-volatile-matter content*

ISO 4618, *Paints and varnishes — Terms and definitions*

ISO 7724-2, *Paints and varnishes — Colorimetry — Part 2: Colour measurement*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
spreading rate
surface area that can be covered by a given quantity of coating material to give a dried film of requisite thickness

NOTE 1 It is expressed in m^2/l or m^2/kg.

NOTE 2 See also practical spreading rate and theoretical spreading rate.

[ISO 4618]

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3.2
practical spreading rate
spreading rate which, in practice, is obtained on the particular substrate being coated

[ISO 4618]

3.3
theoretical spreading rate
spreading rate calculated solely from the volume of non-volatile matter

[ISO 4618]

4 Principle

For method A, coated transparent polyester foils are fixed over a black and white glass panel. The tristimulus values of the coated foils are measured over the black and the white areas. The contrast ratio is calculated as a percentage for each coated foil.

For method B, black and white charts are coated. The tristimulus values of each coated chart are measured over the black and the white areas. The contrast ratio is calculated as a percentage for each coated chart.

5 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

5.1 Substrate, conforming to the requirements of 5.1.1 or 5.1.2.

5.1.1 Colourless transparent polyester foil (method A), untreated, between 30 μm and 50 μm in thickness and of dimensions not less than 100 mm × 150 mm.

NOTE Thicker foils may be used by agreement between the interested parties.

5.1.2 Black and white charts (method B), all the same size and measuring at least 100 mm × 200 mm, printed and varnished to give adjacent black and white areas readily wetted by, but impervious to, solvent- or water-thinned paints.

The black and white areas shall have dimensions not less than 80 mm × 80 mm. The tristimulus value Y of the white areas of the charts shall be 80 ± 2 when measured over a white area using a reflectometer or spectrometer complying with 5.3, and that of the black area shall not be greater than 5, unless otherwise agreed.

To avoid errors due to variation from one batch of charts to another, the charts used for the test shall come from the same batch.

5.2 Film applicators, comprising a series of film applicators giving a range of uniform films of wet thicknesses usually ranging from 50 μm to 100 μm. The film laid down shall be at least 70 mm wide, with an area of uniform thickness measuring not less than 60 mm × 60 mm, regardless of the substrate used.

NOTE The application of uniform films is facilitated by the use of automatic applicators, which are recommended.

5.3 Reflectometer or spectrophotometer, to measure the tristimulus value Y, preferably for D65 standard illuminant, with an accuracy of 0,3 %.

It is recognized that the relative geometrical arrangement of the illumination beam and the light detector can affect the measurement of Y, but it is considered that variations arising from this factor in commercial reflectometers should be considerably less than the reproducibility figure stated in Clause 8. In the event of dispute, diffuse/8° geometry, including surface reflection, shall be used. Surface reflection shall be taken into account by reducing the measured tristimulus value Y mathematically by 4 (see also ISO 7724-2).

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5.4 Test panels, comprising black and white glass panels, at least 6 mm thick, each with a plane, polished surface measuring at least 80 mm × 80 mm. The tristimulus value Y of the white area shall be 80 ± 2 when measured using a reflectometer or spectrometer conforming to the requirements of 5.3, and that of the black area not more than 5.

Both the black and the white areas shall be coated on the back and edges with black paint or adhesive tape to exclude light reflected from the back.

5.5 Syringe, capable of delivering 2 ml or 4 ml to the nearest ± 0,05 ml.

5.6 Analytical balance, capable of weighing to the nearest 0,1 mg.

6 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

Examine and prepare the sample for testing, as described in ISO 1513.

7 Procedure

7.1 Preparation of substrate

7.1.1 Method A (polyester foil)

Retain, for use in 7.5.1, an uncoated sample of polyester foil from an area adjacent to the surface to be coated.

Prepare the polyester foil for coating by one of the following procedures.

Either

a) Spread it on a flat glass panel, at least 6 mm thick, which has first been moistened with a few drops of white spirit just sufficient to hold the foil in position by surface tension. Ensure that none of the liquid wets the upper surface of the foil and that no air bubbles are trapped under it.

Or

b) Fix it at one end and lay it over a flat rubber block (where spiral applicators are to be used).

7.1.2 Method B (black and white charts)

Store the black and white substrate charts, in single thickness, under the test conditions [(23 ± 2) °C and a relative humidity of (50 ± 5) %] for at least 24 h before coating; handle them at all times by the edges to avoid finger marks on the areas to be coated. Weigh, to the nearest 1 mg, six charts for coating, and two charts to be kept as blank controls.

Prepare the charts for coating by one of the following procedures:

a) fix one end, by clips or adhesive tape, to a flat glass panel at least 6 mm thick;

b) use a vacuum suction plate, which shall be flat to within ± 2 µm;

c) fix it at one end and lay it over a flat rubber block (where spiral applicators are to be used).

7.2 Preparation of coated charts or foils

Immediately before application, mix the paint thoroughly by vigorous stirring to break down any thixotropic structure, taking care not to incorporate air bubbles.

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Using the syringe (5.5), apply about 2 ml to 4 ml of paint, depending on the film thickness required, in a line across one end of the chart or polyester foil and spread it immediately by drawing down a suitable applicator at a steady speed to give a uniform layer. Prepare duplicate films with each of three different applicators, chosen to give a range of wet film thicknesses usually ranging from 50 μm to 100 μm.

Maintain the coated charts or foils in a horizontal position until dry, for example by taping the edges to a flat substrate.

NOTE The drying time (and/or stoving conditions) depends on the type of paint material being tested, and should be agreed by the interested parties.

7.3 Conditioning

Unless otherwise agreed, keep the dried coated charts or foils and the blank charts or foils at (23 ± 2) °C and a relative humidity of (50 ± 5) % for at least 24 h before the measurements of the tristimulus values Y are made.

7.4 Measurement of tristimulus value Y

7.4.1 Method A (polyester foil)

Fix each coated foil over a black and white glass panel, introducing a few drops of white spirit between the underside of the foil and the glass to ensure optical contact. Measure the tristimulus value Y of each coated foil at a minimum of four positions over the black (Y_b) and white (Y_w) areas, and calculate the mean tristimulus values Y_b and Y_w respectively. Then calculate the contrast ratio Y_b/Y_w as a percentage for each coated foil.

7.4.2 Method B (black and white charts)

Measure the tristimulus values of each coated chart at a minimum of four positions over both the black and the white areas of each chart and calculate the mean tristimulus values Y_b and Y_w respectively. Then calculate the contrast ratio Y_b/Y_w as a percentage for each coated chart.

7.5 Determination of the mass per unit area of the dry coating

7.5.1 Method A (polyester foil)

Remove the coated foil from the glass panel, wipe the foil to remove any traces of white spirit and allow to dry.

Cut an equal area with dimensions at least 60 mm × 60 mm, for example by means of a precision die stamp, from the centre of each coated polyester foil and two samples from the uncoated (blank) polyester foil.

Weigh the detached pieces to the nearest 1 mg.

Calculate the mean mass for the coated polyester foil and the mean mass for the two samples of uncoated polyester foil.

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Calculate the mass per unit area of the dry coating, m'', in grams per square millimetre, using Equation (1):

$$m'' = \frac{m_2 - m_1}{A} \quad (1)$$

where

m_1 is the mean mass, in grams, of the two samples of uncoated polyester foil;

m_2 is the mean mass, in grams, of the six samples of coated polyester foil;

A is the area, in square millimetres, of the area cut out in each case.

7.5.2 Method B (black and white charts)

Cut equal areas, for example by means of a precision die stamp, with dimensions at least 60 mm × 60 mm, from the centres of the blank and the coated charts. Weigh the detached pieces to the nearest 1 mg.

Calculate the mass per unit area of the dry coating, m'', in grams per square millimetre, using Equation (2):

$$m'' = \frac{m_4 - \left(\frac{m_3 \times m_2}{m_1}\right)}{A} \quad (2)$$

where

m_1 is the mean mass, in grams, of the two blank control charts;

m_2 is the mean initial mass, in grams, of the other six charts before coating;

m_3 is the mean mass, in grams, of the cut out portions of the blank control charts;

m_4 is the mean mass, in grams, of the cut out portions of the coated charts;

A is the area, in square millimetres, of the area cut out in each case.

NOTE This procedure eliminates the effect of changes in the masses of the charts due to variations in moisture content if it can be assumed that blank and coated charts change equally.

7.6 Calculation of theoretical wet film thickness and practical spreading rate

7.6.1 General

To calculate the wet film thickness from the mass per unit area of the dry coating, it is necessary to know both the density of the coating material, as obtained by using one of the methods given in ISO 2811-1 to ISO 2811-4, and the non-volatile-matter content using the method given in ISO 3251.

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7.6.2 Theoretical wet film thickness

Calculate the theoretical thickness of the wet coating material, t_W, in millimetres, using Equation (3).

$$t_W = \frac{m''}{\rho \times NV} \times 10^5 \quad (3)$$

where

m'' is the mass per unit area of the dry coating, in grams per square millimetre;

ρ is the density of the coating material, in grams per millilitre;

NV is the non-volatile-matter content, as a percentage by mass.

7.6.3 Practical spreading rate

7.6.3.1 Method A (polyester foil)

The practical spreading rate, S_P, in square metres per litre, is the reciprocal of the theoretical wet film thickness, in millimetres, and is given by Equation (4).

$$S_P = \frac{1}{t_W} = \frac{\rho \times NV}{m''} \times 10^{-5} \quad (4)$$

and, using Equation (1) for mass per unit area of the dry coating,

$$S_P = \frac{A \times \rho \times NV}{m_2 - m_1} \times 10^{-5} \quad (5)$$

7.6.3.2 Method B (black and white charts)

The practical spreading rate, S_P, in square metres per litre, is the reciprocal of the theoretical wet film thickness, in millimetres, and is given by Equation (6).

$$S_P = \frac{1}{t_W} = \frac{\rho \times NV}{m''} \times 10^{-5} \quad (6)$$

and, using Equation (2) for mass per unit area of the dry coating,

$$S_P = \frac{A \times \rho \times NV}{m_4 - \left(\frac{m_3 \times m_2}{m_1}\right)} \times 10^{-5} \quad (7)$$

8 Precision

8.1 Repeatability (r)

The value below which the absolute difference between two single test results, each the mean of duplicates, obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method may be expected to lie with a 95 % probability is 1 % using method A, or 2 % using method B.

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8.2 Reproducibility (R)

The value below which the absolute difference between two test results, each the mean of duplicates, obtained on identical material by operators in different laboratories using the standardized test method may be expected to lie with 95 % probability is 2 % using method A, or 4 % using method B.

9 Test report

The test report shall contain at least the following information:

a) all information necessary for identification of the sample tested;

b) a reference to this part of ISO 6504 (ISO 6504-3:2006);

c) the method used (A or B) and

if method A was used, the values Y_b and Y_w for the polyester foil,

if method B was used, the corresponding values for the charts;

d) if method A was used, the thickness of the polyester foils;

e) the drying time and/or stoving conditions;

f) the values of the paint density and the non-volatile-matter content used in calculating the test result;

g) the results of the test, expressed as the contrast ratio determined for a spreading rate of 20 m^2/l and calculated as specified in Clause 7;

h) any deviations from the procedure specified;

i) any unusual features (anomalies) observed during the test;

j) the date of the test.

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Part 3: Determination of contrast ratio of light-coloured paints at a fixed spreading rate

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 6504-3 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 6504-3:1998), which has been technically revised.

The main technical changes are:

a) the unit of density of the wet coating material, used in Equation (3) in 7.6.2, has been corrected;

b) the surface density (ρ_A) has been changed to mass per unit area of the dry coating (m'').

The opportunity has also been taken to revise the text editorially.

ISO 6504 consists of the following parts, under the general title *Paints and varnishes — Determination of hiding power*:

— *Part 1: Kubelka-Munk method for white and light-coloured paints*

— *Part 3: Determination of contrast ratio of light-coloured paints at a fixed spreading rate*

The subject originally intended to be covered by Part 2 is currently covered by ISO 2814, *Paints and varnishes — Comparison of contrast ratio (hiding power) of paints of the same type and colour*.

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Introduction

Two techniques are available for test film preparation and measurement when determining the contrast ratio of paints:

a) application to colourless, transparent polyester foil, the coated foil being subsequently placed in turn over black and white glass panels;

b) direct application to black and white charts, for example Morest charts.

Because different operators using the same draw-down device will obtain paint films differing significantly in thickness, an absolute method for the determination of opacity is required. Collaborative trials between groups of experts from a number of countries have shown that reproducible results can be obtained by determination of the contrast ratio corresponding to a precisely fixed spreading rate by interpolation between measurements at two or more measured film thicknesses. The spreading rate selected in this part of ISO 6504 is 20 m^2/l (wet film thickness 50 µm), which is considered to be an average for brush application of a free-flowing paint on a smooth, non-porous surface. However, for particular types of paint normally used at other film thickness ranges, for example industrial enamels and printing inks, the interested parties may agree on another spreading rate.

Further collaborative trials have indicated that higher reproducibility is obtained with films spread on polyester foil than with films spread on a black and white chart, although the latter technique is simpler to operate. This part of ISO 6504 provides for both these options.

The methods are based on the observation that contrast ratio is an approximately linear function of reciprocal spreading rate, over a restricted film thickness range which also corresponds to that used for normal application of white or light-coloured paints. It is thus possible to interpolate graphically or by computation, with satisfactory accuracy, between results obtained with films of different thicknesses.

Because the wet film thickness is not always determined directly, the methods specified in this part of ISO 6504 involve the determination of dry film mass per unit area and calculation of the corresponding wet film thickness. In this latter calculation, values for wet paint density and percentage of non-volatile-matter content are required. Determination of these values by the methods specified in the relevant International Standards has been stipulated. However, it is recognized that, for certain types of paint, the non-volatile-matter determination in accordance with ISO 3251 does not correspond exactly to the mass changes in a film during drying under the conditions of the present test method. Any errors introduced into results by this discrepancy should be common to all laboratories, however, and should not affect comparisons of paints of similar types.

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