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General methods of test for pigments and extenders —

Part 25: Comparison of the colour, in full-shade systems, of white, black and coloured pigments — Colorimetric method

Méthodes générales d'essai des pigments et matières de charge —

Partie 25: Comparaison, dans les systèmes monopigmentaires, de la couleur des pigments blancs, noirs et colorés — Méthode colorimétrique

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Contents

Page

Foreword

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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).

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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 256, *Pigments, dyestuffs and extenders*.

This second edition cancels and replaces the first edition (ISO 787-25:1993), which has been technically revised. The main changes compared to the previous edition are as follows:

— Clause 3 has been revised and terms and definitions for full shade, mass tone and mass tone system have been added/revised;

— the normative references have been updated;

— the text has been editorially revised.

A list of all parts in the ISO 787 series can be found on the ISO website.

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.

General methods of test for pigments and extenders —

Part 25: Comparison of the colour, in full-shade systems, of white, black and coloured pigments — Colorimetric method

1 Scope

This document specifies a general test method for comparing the colour, in full-shade systems, of white, black or coloured pigments with that of an agreed reference pigment, using a colorimetric procedure.

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 787-9, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension*

ISO 787-24, *General methods of test for pigments and extenders — Part 24: Determination of relative tinting strength of coloured pigments and relative scattering power of white pigments — Photometric methods*

ISO 2114, *Plastics (polyester resins) and paints and varnishes (binders) — Determination of partial acid value and total acid value*

ISO 3219, *Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate*

ISO 3262-20, *Extenders for paints — Specifications and methods of test — Part 20: Fumed silica*

ISO 4629-1, *Binders for paints and varnishes — Determination of hydroxyl value — Part 1: Titrimetric method without using a catalyst*

ISO 8780-6, *Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics — Part 6: Dispersion using a triple-roll mill*

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

ISO 18314-1, *Analytical colorimetry — Part 1: Practical colour measurement*

ISO 18314-2, *Analytical colorimetry — Part 2: Saunderson correction, solutions of the Kubelka-Munk equation, tinting strength, hiding power*

ISO 18451-1, *Pigments, dyestuffs and extenders — Terminology — Part 1: General Terms*

ISO 18451-2, *Pigments, dyestuffs and extenders — Terminology — Part 2: Classification of colouring materials according to colouristic and chemical aspects*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18451-1, ISO 18451-2 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
full shade
colour of a mass tone system in optically infinite (hiding) layer

[SOURCE: ISO 18451-1:2019, 3.41]

3.2
mass tone
colour of a mass tone system in a non-hiding layer

[SOURCE: ISO 18451-1:2019, 3.69]

3.3
mass tone system
pigmented system that contains one pigment only

[SOURCE: ISO 18451-1:2019, 3.70]

4 Principle

The test pigment and an agreed reference pigment are dispersed in a particular test medium, consisting of a mixture of an alkyd resin and fumed silica, using an automatic muller. From the dispersions of the two pigments, specimens on suitable substrates are prepared. The tristimulus values of the specimens are measured as described in ISO 18314-1 and from these, the appropriate colour characteristics (relative hue and amount of relative hue for black and white pigments). Lightness, hue, chroma and total colour difference for coloured pigments are calculated as described in ISO 11664-4.

5 Materials

5.1 Alkyd resin (binder)

The alkyd resin shall consist of 63 % (mass fraction) linseed oil, 23 % (mass fraction) phthalic anhydride and 14 % (mass fraction) trimethylol propane, and shall comply with the following requirements:

		Test method
Acid value	max. 15 mg KOH/g	ISO 2114
Viscosity (of the product as delivered)	7 Pa · s to 10 Pa · s	ISO 3219
Hydroxyl value	30 mg to 50 mg KOH/g	ISO 4629-1

5.2 Fumed silica

The fumed silica shall comply with the following requirements:

		Test method
Specific surface area (BET)	175 m^2/g to 225 m^2/g	ISO 3262-20
pH value of a 4 % dispersion in water	3,6 to 4,5	ISO 787-9

Fumed silica is necessary to avoid flocculation and to control the flow properties of the pigmented system.

5.3 Preparation of the test medium

The preferred test medium (see Table 1 and Table 2 for quantities) is prepared as follows.

Mix 97 parts by mass of alkyd resin (5.1) and 3 parts by mass of fumed silica (5.2) well. Take care that no silica is lost by evolution of dust. Disperse the mixture twice on a triple-roll mill according to ISO 8780-6.

Another test medium may be agreed between the interested parties. This shall be indicated in the test report.

Table 1 — Recommended quantities of white pigment and test medium

Pigment (Density) g/ml	Mass of pigment g	Volume of test medium (5.3) ml
Titanium dioxide (ρ = 4,0)	4,0	3,0
Zinc sulfide (ρ = 4,0)	4,0	2,8
Zinc oxide (zinc white) (ρ = 5,8)	5,0	2,6

Table 2 — Recommended quantities of coloured and black pigments and test medium

Pigment group (see 8.2.3)	Mass of pigment g	Volume of test medium (5.3) ml
a	3,0	1,5
b	1,0	1,5
c	0,5	1,5

6 Apparatus

Ordinary laboratory apparatus and glassware, together with the following.

6.1 Photometer

6.1.1 For coloured and white pigments

Spectrometer or tristimulus colorimeter as specified in ISO 18314-1.

6.1.2 For black pigments

Spectrometer or tristimulus colorimeter as specified in ISO 18314-1 and complying with the following requirements.

a) Accuracy

A spectrometer shall provide reflectance values to five decimal places; a tristimulus colorimeter shall provide tristimulus values to three decimal places.

b) Calibration and zero adjustment

The instrument should preferably be such that it can be adjusted by using a suitable black standard so that the data for the tristimulus values are close to zero. If direct adjustment cannot be performed, the readings for the black standard shall be subtracted from the values for the test specimens.

The black standard used for the zero adjustment shall be a highly efficient light trap as shown in Figure 1 and having the following dimensions:

— A ≥ diameter of instrument sample port +5 mm;

— B ≥ 80 mm;

— C ≥ 70 mm.

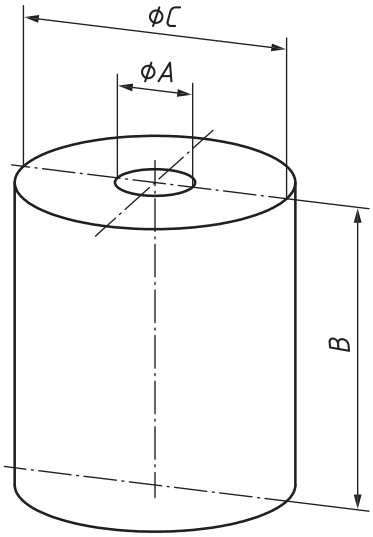

Figure 1 — Black standard

The black standard shall have a non-gloss black interior and the bottom shall be covered with a mat black coating.

c) Standard deviation of tristimulus values

The standard deviation σ_r for consecutive measurements of tristimulus values shall be < 0,005. It shall be calculated from 20 measurements of tristimulus values without changing the position of a test specimen having a tristimulus value Y of about 0,5.

6.2 Substrate, of minimum dimensions 150 mm × 50 mm, uniform, non-fluorescent, compatible with the binder to be used and appropriate to the method of colour comparison.

Steel panels, contrast cards, lacquered cardboard sheets, aluminium-covered cards, standard art paper or glass slides may be used. If a glass slide is used, it shall be clear and colourless, and of the same thickness for the reference and test specimens.

6.3 Film applicator, for the application of the pastes of the test pigment and the agreed reference pigment to the substrate.

6.4 Stencil card (for covering specimens that are still moist when presented for measurement), approximately 0,5 mm thick, with a circular hole of diameter slightly greater than the size of the measure orifice of the spectrometer or tristimuius colorimeter (6.1), or glass plates of sufficient size to cover the specimen, coplanar, polished, colourless, about 1 mm thick.

6.5 Automatic muller, with ground-glass plates, preferably water-cooled. The plates shall be of diameter 180 mm to 250 mm and such that a known, selectable force of up to 1 kN may be applied to them. The driven plate shall be capable of rotating speed at between 70 min^{-1} and 120 min^{-1} and the apparatus shall have an arrangement for pre-setting the number of revolutions in multiples of 25.

6.6 Spatula, with flexible steel or plastic blade.

7 Sampling

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

8 Procedure

8.1 General

Take care that the results are not influenced by any temperature increase during the grinding operation. This applies particularly if the plates of the automatic muller are not water-cooled. If any influence is suspected, carry out preliminary tests to check this.

Precondition of new muller plates occurs by milling a pigment in a suitable binder for 1 000 revolutions with a load applied to the plates. Remove and discard the paste.

Before use, check that the surfaces of each plate have an even, matt appearance and are free from notch marks and polished areas.

8.2 Test portion

8.2.1 Generals

Take a sufficient quantity of the pigment so that, when mixed with the appropriate amount of test medium (5.3), the resulting paste extends almost to the edges of the muller plates. Weigh this test portion to the nearest 1 mg.

8.2.2 White pigments

Preferably use the recommended quantities of pigment and test medium given in Table 1 (see also 8.2.2, NOTE 1 and NOTE 2.)

8.2.3 Coloured and black pigments

The mass ratio of pigment to binder depends not only on the oil absorption of the pigment but also on the viscosity of the mix during the milling operation. As a first step, all pigments can be allocated to one of the following three groups:

Group a: pigments of low binder demand — pigment concentration 65 % (mass fraction)

Group b: pigments of medium binder demand — pigment concentration 40 % (mass fraction)

Group c: pigments of high binder demand — pigment concentration 25 % (mass fraction)

In order to give approximately 2 ml of mixture in each case, preferably use the recommended quantities for the appropriate group as given in Table 2 (see also NOTE 1 and NOTE 2).

NOTE 1 If the pigment/binder mix chosen is found to be too stiff or fluid for use on the muller, one of the other pairs of quantities given in Table 1 or Table 2 can be used as appropriate.

NOTE 2 If the diameter of the muller plates is near the maximum of the range specified in 6.5, it might be necessary to increase the quantities specified in order to reduce wear on the plates.

8.3 Preparation of pigment dispersions

Take the appropriate quantities of the test medium and the agreed reference pigment (8.2.1 or 8.2.2). Place the test medium in the centre of the lower plate of the automatic muller (6.5). Sprinkle the pigment into the test medium and mix together, using the minimum effort, with the aid of the spatula (6.6). Distribute the paste at several points at a distance of about 35 mm from the centre of the lower plate or spread in the form of a ring with an internal diameter of 40 mm and an external diameter of 100 mm.

It is advisable to lay a paper ring of the requisite shape as a pattern beneath the lower plate.

Clean the spatula as much as possible by wiping it on the upper plate of the muller.

Close the plates of the muller and grind the mixture in stages of 50 revolutions with the highest practicable load or a load agreed between the interested parties. After each stage, collect the paste with the spatula from both plates and spread it as described above on the lower plate, wiping the spatula on the upper plate as before. After the required number of revolutions, remove and store the paste in a suitable vessel and then clean the muller plates and spatula.

Grind the mixture for the same total number of revolutions as required for the full determination of the dispersibility in accordance with ISO 787-24, to ensure complete dispersion of the pigment. Indicate the number of revolutions in the test report.

Take a similar amount of the test pigment and prepare a paste in the same way.

Alternative equipment for the dispersion of pigments may be agreed between the interested parties. This shall be indicated in the test report.

8.4 Preparation of test specimens

8.4.1 General

The preparation of the test specimens depends on the method of application, the substrate and the film thickness, which shall be chosen according to the intended application of the particular pigmented system.

Apply the test pigment dispersion and the agreed reference pigment dispersion by the same method over a width of at least 40 mm.

Measure wet samples as soon as possible after application.

8.4.2 White pigments

Spread the paste with the film applicator (6.3) on to the substrate (6.2), using a clearance of 150 µm to 200 µm for titanium dioxide and zinc sulfide (100 %) pigments, and of 500 µm for lithopone and zinc oxide (zinc white) pigments.

8.4.3 Coloured and black pigments

8.4.3.1 Hiding layers for evaluating differences in full-shade colour

Apply the test pigment dispersion and the agreed reference pigment dispersion in a hiding layer.

NOTE A layer is considered to be a hiding layer when, on visual inspection of a film laid on a black and white contrasting test substrate, the contrast is no longer visible.

8.4.3.2 Non-hiding layers for evaluating differences

Apply the test pigment dispersion and the agreed reference pigment dispersion side by side in one and the same application process (if possible) and in the same film thickness.

8.5 Measurement

Measure the tristimulus values of the specimens prepared from the test pigment dispersion and of the agreed reference pigment dispersion as described in ISO 18314-1, using the appropriate geometry (see ISO 18314-1 and ISO 18314-2).

The aim of this document is to test pigments, and differences in the surface reflectance of specimens to be compared shall therefore not be allowed to influence the results. With the exception of very dark

samples, this can be achieved by using the 8/*d* or *d*/8 geometry with specular reflection included. With high-gloss specimens, subtract the value of surface reflection from the measurement to enable a better correlation with the visual evaluation. 45/0 or 0/45 geometry can only be used with high-gloss or completely matt specimens. With very dark specimens (for example those prepared from carbon blacks or in the case of transparent pigments), care shall be taken that differences in gloss or surface structure do not affect the reflectance values. In some cases, it is advisable to apply the dispersions to a glass plate and to measure through this glass plate, using the 45/0 or *d*/8 geometry with specular reflection excluded.

9 Expression of results

From the measured values, obtain the colour characteristics indicated in 9.1 or 9.2 respectively, using the formulae given in ISO 18314-2.

9.1 White pigments and black pigments

9.1.1 Relative hue

Calculate the values using Formulae (1) and (2):

$$\Delta a^* = a^*_T - a^*_R \tag{1}$$

and

$$\Delta b^* = b^*_T - b^*_R \tag{2}$$

where

a^*, b^* are the CIELAB a^*, b^* coordinates;

Δa^*,Δb^* are the CIELAB a^*, b^* difference;

R is the reference specimen;

T is the test specimen.

Determine the relative hue from the signs of Δa^* and Δb^* and the value of $\left|\Delta b^* / \Delta a^*\right|$ (the absolute value of the ratio). Express the relative hue by the appropriate colour taken from Table 3.

Table 3 — Description of relative hue

Sign of Δb^*	**Sign of Δa^***		$\left\|\Delta b^* / \Delta a^*\right\|$
	–	**+**	
+	yellow (Y)		> 2,5
	yellow-green (YG)	yellow-red (YR)	0,4 to 2,5
+ or –	green (G)	red (R)	< 0,4
–	blue-green (BG)	blue-red (BR)	0,4 to 2,5
	blue (B)		> 2,5