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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

Reference number
ISO 787-25:1993(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 787-25 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Sub-Committee SC 2, *Pigments and extenders*.

ISO 787 consists of the following parts, under the general title *General methods of test for pigments and extenders*:

- — *Part 1: Comparison of colour of pigments*
- — *Part 2: Determination of matter volatile at 105 °C*
- — *Part 3: Determination of matter soluble in water — Hot extraction method*
- — *Part 4: Determination of acidity or alkalinity of the aqueous extract*
- — *Part 5: Determination of oil absorption value*
- — *Part 7: Determination of residue on sieve — Water method — Manual procedure*
- — *Part 8: Determination of matter soluble in water — Cold extraction method*
- — *Part 9: Determination of pH value of an aqueous suspension*

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- *Part 10: Determination of density — Pyknometer method*
- *Part 11: Determination of tamped volume and apparent density after tamping*
- *Part 13: Determination of water-soluble sulphates, chlorides and nitrates*
- *Part 14: Determination of resistivity of aqueous extract*
- *Part 15: Comparison of resistance to light of coloured pigments of similar types*
- *Part 16: Determination of relative tinting strength (or equivalent colouring value) and colour on reduction of coloured pigments — Visual comparison method*
- *Part 17: Comparison of lightening power of white pigments*
- *Part 18: Determination of residue on sieve — Mechanical flushing procedure*
- *Part 19: Determination of water-soluble nitrates (Salicylic acid method)*
- *Part 21: Comparison of heat stability of pigments using a stoving medium*
- *Part 22: Comparison of resistance to bleeding of pigments*
- *Part 23: Determination of density (using a centrifuge to remove entrained air)*
- *Part 24: Determination of relative tinting strength of coloured pigments and relative scattering power of white pigments — Photometric methods*
- *Part 25: Comparison of the colour, in full-shade systems, of white, black and coloured pigments — Colorimetric method*
- *Part 26: Determination of relative tinting strength and remaining colour difference on reduction — Colorimetric method*

Parts 13, 14 and 17 are bound together as one document. Parts 6, 12 and 20 have been withdrawn.

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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

1 Scope

This part of ISO 787 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.

NOTE 1 When the general method specified in this part of ISO 787 is applicable to a given pigment, only a cross-reference to ISO 787-25 needs to be included in the International Standard giving the specification for that pigment, indicating any detailed modification that may be needed in view of the special properties of the material in question. Only when the general method in this part of ISO 787 is not applicable to a particular material is a different photometric method for comparison of colour to be specified.

ISO 787-1:1982, *General methods of test for pigments and extenders — Part 1: Comparison of colour of pigments*, describes a general test method for visual comparison of the colour of pigments.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 787. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 787 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 787-9:1981, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension.*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

ISO 3219:1993, *Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate.*

ISO 3682:1983, *Binders for paints and varnishes — Determination of acid value — Titrimetric method.*

ISO 4629:1978, *Paint media — Determination of hydroxyl value — Titrimetric method.*

ISO 7724-1:1984, *Paints and varnishes — Colorimetry — Part 1: Principles.*

ISO 7724-2:1984, *Paints and varnishes — Colorimetry — Part 2: Colour measurement.*

ISO 7724-3:1984, *Paints and varnishes — Colorimetry — Part 3: Calculation of colour differences.*

ISO 8780-6:1990, *Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics — Part 6: Dispersion using a triple-roll mill.*

ASTM D 16-1991, *Standard terminology relating to paint, varnish, lacquer and related products.*

ASTM D 387-1986, *Standard test method for color and strength of color pigments with a mechanical muller.*

ASTM D 3022-1984, *Standard test method for color and strength of color pigments by use of a miniature sandmill.*

3 Definitions

For the purposes of this part of ISO 787, the following definitions apply.

3.1 full-shade system: A pigmented system that contains one pigment only.

3.2 full-shade colour: The colour of a full-shade system that is applied in a hiding layer (optically infinite). (See also note 3.)

NOTE 2 Hiding cannot be achieved with some coloured pigments of very high transparency.

3.3 mass tone: The colour of a full-shade system that is not applied in a hiding layer, for example on a white test substrate. (See also notes 3 and 4.)

NOTES

3 The definitions given in 3.2 and 3.3 are in conflict with those in ASTM D 16 and with the usage of "mass color" in ASTM D 387 and ASTM D 3022.

4 With pigmented systems, it is possible to produce a number of different mass tones, depending on the thickness of the applied layer and the properties of the test substrate. Therefore, a mass tone is only clearly defined if the preparation, the composition, the application technique and the film thickness of the pigmented system as well as the substrate are defined.

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 7724-2 and from these the appropriate colour characteristics (relative hue and amount of chromaticity difference for black and white pigments; lightness, hue, chroma and total colour difference for coloured pigments) are calculated as described in ISO 7724-3.

5 Materials

5.1 Alkyd resin (binder)

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

		Test method
Acid value	max. 15 mg KOH/g	ISO 3682
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

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	to be agreed between the interested parties
pH value of a 4 % dispersion in water	3,6 to 4,5	ISO 787-9

NOTE 5 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 tables 1 and 2 for quantities) is prepared as follows:

Mix well 97 parts by mass of alkyd resin (5.1) and 3 parts by mass of fumed silica (5.2). Take care that no silica is lost by evolution of dust. Disperse the mixture twice on a triple-roll mill (see 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 ($\rho = 4,0$)	4,0	3,0
Zinc sulfide ($\rho = 4,0$)	4,0	2,8
Zinc oxide (zinc white) ($\rho = 5,8$)	5,0	2,6

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

Pigment group (see 8.1.2)	**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 7724-2.

6.1.2 For black pigments

Spectrometer or tristimulus colorimeter as specified in ISO 7724-2 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 reference and 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

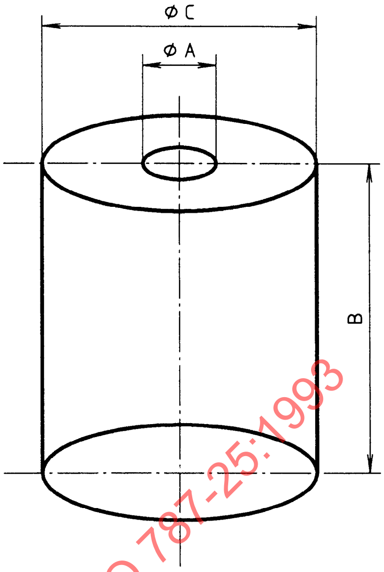

Figure 1 — Black standard

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

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 sample port of the spectrometer or tristimulus colorimeter (6.1), or glass plates of sufficient size to cover the specimen, plane parallel, 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 at between 70 r/min and 120 r/min 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 842.

8 Procedure

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 new muller plates 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 score marks and polished areas.

8.1 Test portion

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.

8.1.1 White pigments

Preferably use the recommended quantities of pigment and test medium given in table 1. (See also notes 6 and 7.)

8.1.2 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 % (*m/m*)

Group b: pigments of medium binder demand — pigment concentration 40 % (*m/m*)

Group c: pigments of high binder demand — pigment concentration 25 % (*m/m*)

In order to give approximately 2 ml of mix in each case, preferably use the recommended quantities for the appropriate group as given in table 2. (See also notes 6 and 7.)

NOTES

6 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 2 may be used as appropriate.

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

8.2 Preparation of pigment dispersions

Take the appropriate quantities of the test medium and the agreed reference pigment (8.1.1 or 8.1.2). 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.

NOTE 8 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 receptacle 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 assure 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.

8.3 Preparation of test specimens

8.3.1 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.

8.3.2 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.

8.3.3 Coloured and black pigments

8.3.3.1 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 9 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.

8.3.3.2 Non-hiding layers for evaluating differences in mass tone

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.

8.4 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 7724-2, using the appropriate geometry (see ISO 7724-1 and ISO 7724-3).

The aim of this part of ISO 787 is to test pigments, and differences in the surface reflectance of specimens to be compared must not therefore 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 mass tones 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 equations given in ISO 7724-3.

9.1 White pigments and black pigments

9.1.1 Relative hue

Calculate the values

$$\Delta a^* = a^*_{\mathrm{T}} - a^*_{\mathrm{R}}$$

and

$$\Delta b^* = b^*_{\mathrm{T}} - b^*_{\mathrm{R}}$$

where

R refers to the reference specimen;

T refers to the test specimen.

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

Table 3 — Name of relative hue

Sign of Δb^*	**Sign of Δa^***		$\lvert\Delta b^*/\Delta a^*\rvert$
	−	+	
+	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

9.1.2 Amount of chromaticity difference

Calculate the value Δs, by which the amount of chromaticity difference is characterized, using the following equation: