
International Standard



787/24

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

**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**

Méthodes générales d'essai des pigments et matières de charge — Partie 24 : Détermination du pouvoir colorant relatif des pigments colorés et du pouvoir diffusant relatif des pigments blancs — Méthodes photométriques

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Descriptors : paints, pigments, tests, determination, colouring power, photometric method.

The purpose of this International Standard is to establish a series of general test methods for pigments and extenders which are suitable for all or many of the individual pigments and extenders for which specifications might be required. In such cases, a cross-reference to the general method should be included in the International Standard relating to that pigment or extender, with a note of any detailed modification which might be needed in view of the special properties of the product in question.

Technical Committee ISO/TC 35, *Paints and varnishes*, decided that all the general methods should be published as they become available, as parts of a single International Standard, in order to emphasize the relationship of each to the whole series.

The Technical Committee also decided that, where two or more procedures were widely used for determining the same or a similar characteristic of a pigment or extender, there would be no objection to including more than one of them in the ISO series. In such cases it will, however, be essential to state clearly in a specification which method is to be used and, in the test report, which method has been used.

Parts of the series already published are as follows :

[ISO 787-24:1985](https://standards.iteh.ai/catalog/standards/sist/9359337c-0715-44e7-8ce3-7d53071dcb/a/iso-787-24-1985)

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- 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
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- Part 7 : Determination of residue on sieve — Water method — Manual procedure
- Part 8 : Determination of matter soluble in water — Cold extraction method
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- Part 11 : Determination of tamped volume and apparent density after tamping
- Part 13 : Determination of water-soluble sulfates, 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 — Water method — Mechanical flushing procedure
- Part 19 : Determination of water-soluble nitrates — Salicylic acid method
- Part 20 : Comparison of ease of dispersion — Oscillatory shaking 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

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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

0 Introduction

This document is a part of ISO 787, *General methods of test for pigments and extenders*.

1 Scope and field of application

This part of ISO 787 describes photometric methods of test for comparing in the visible spectrum

- a) the tinting strength of two similar (see note 1) coloured pigments dispersed in an alkyd resin without a drier;
- b) the scattering power of two white pigments of the same type dispersed in an alkyd resin without a drier.

These methods of test provide an instrumental alternative to those described in ISO 787/16 and ISO 787/17 respectively, avoiding the necessity of visual matching.

NOTES

- 1 This International Standard is not appropriate for the comparison of coloured pigments that on reduction differ greatly in colour.
- 2 When one of these general methods is applicable to a given pigment, only a cross-reference to the appropriate method should be included in the International Standard relating to that pigment, indicating any detailed modification which may be needed in view of the special properties of the product in question. Only when the appropriate method in this part of ISO 787 is not applicable to a particular product should different photometric methods for determination of relative tinting strength and determination of relative scattering power be specified.

2 References

ISO 591, *Titanium dioxide pigments for paints*.

ISO 787, *General methods of test for pigments and extenders —*

Part 2 : Determination of matter volatile at 105 °C.

Part 9 : Determination of pH value of an aqueous suspension.

Part 10 : Determination of density — Pyknometer method.

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 23 : Determination of density (using a centrifuge to remove entrained air).

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

ISO 1524, *Paints and varnishes — Determination of fineness of grind.*

ISO 3219, *Plastics — Polymers in the liquid, emulsified or dispersed state — Determination of viscosity with a rotational viscometer working at defined shear rate.*

ISO 3262, *Extenders for paints.*

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

ISO 4629, *Binders for paints and varnishes — Determination of hydroxyl value — Titrimetric method.*¹⁾

ISO 4652, *Rubber compounding ingredients — Carbon black — Determination of specific surface area — Nitrogen adsorption methods.*

ISO 6209, *Rubber compounding ingredients — Carbon black — Determination of solvent extractable material.*

1) In course of preparation. (Revision of ISO 4629-1978.)

3 Definitions

3.1 For coloured and black pigments

3.1.1 tinting strength : The ability of a pigment (see the note) to absorb incident light, thereby having the power to colour or darken, for example, a white paint in which it is incorporated.

NOTE — Although the properties tested are strictly those of the pigmented binder, absorption and scattering by the binder are relatively small and it is assumed in this International Standard that the material under test is the dispersed pigment.

3.1.2 spectral absorption coefficient $K(\lambda)$: The fraction of the diffusely incident spectral radiant flux of wavelength λ that is absorbed in an elementary layer within a material, divided by the thickness of the layer (Kubelka-Munk analysis).

NOTE — $K(\lambda)$ is a measure of the tinting strength of coloured pigments in a material and is expressed in units of reciprocal film thickness.

3.1.3 absorbance index $K_p(\lambda)$: The spectral absorption coefficient of the pigmented binder divided by the pigment concentration C_m :

$$K_p(\lambda) = \frac{K(\lambda)}{C_m} \quad \dots (1)$$

where C_m is the pigment concentration, for example, expressed as the ratio by mass of pigment to binder.

3.1.4 relative tinting strength $K_r(\lambda)$: The ratio of the absorbance index of the test sample $K_{p1}(\lambda)$ to that of an agreed reference pigment $K_{p2}(\lambda)$, expressed as a percentage :

$$K_r(\lambda) = \frac{K_{p1}(\lambda)}{K_{p2}(\lambda)} \times 100 \quad \dots (2)$$

3.2 For white pigments

3.2.1 scattering power : The ability of a pigment (see the note to 3.1.1) to diffuse incident light, thereby having the power to confer opacity and lightness to, for example, a paint in which it is incorporated.

3.2.2 spectral scattering coefficient $S(\lambda)$: The net transfer of spectral radiant flux of wavelength λ in the outward direction from an elementary layer within a body of material illuminated from outside, divided by the product of the thickness of the layer and the difference between the magnitude of the flux in the two directions through the layer (Kubelka-Munk analysis).

NOTE — $S(\lambda)$ is a measure of the scattering power of white pigments in a material and is expressed in units of reciprocal film thickness.

3.2.3 scattering index $S_p(\lambda)$: The spectral scattering coefficient of the pigmented binder divided by the pigment concentration C_m (see 3.1.3) :

$$S_p(\lambda) = \frac{S(\lambda)}{C_m} \quad \dots (3)$$

3.2.4 relative scattering power $S_r(\lambda)$: The ratio of the scattering index of the test sample $S_{p3}(\lambda)$ to that of an agreed reference pigment $S_{p4}(\lambda)$, expressed as a percentage :

$$S_r(\lambda) = \frac{S_{p3}(\lambda)}{S_{p4}(\lambda)} \times 100 \quad \dots (4)$$

3.3 reflectivity ρ_∞ : The reflectance of a paste or a paint film of such thickness that further increase in thickness gives no further change in reflectance.

The reflectivity ρ_∞^* , corrected according to equation (10), is related to K/S by the equation

$$\frac{K}{S} = \frac{(1 - \rho_\infty^*)^2}{2 \rho_\infty^*} \quad \dots (5)$$

Values of K/S as a function of 100 ρ_∞ or 100 R_∞ are given in annex B.

NOTES

1 Kubelka-Munk analysis is strictly applicable only to monochromatic radiation, as indicated in equations (1) to (4). In practice, however, correct results are often also obtained if the mean value for wider wavebands is taken, as is the case when filters are used. Hence, in the subsequent equations the symbol λ is omitted.

2 In a dispersion containing a coloured or black pigment and a white pigment, K is assumed to be characteristic of the coloured or black pigment and S of the white pigment.

3.4 reflectance factor R_∞ : The ratio of the radiant flux reflected in the directions within a given cone by a paste or a paint film to that reflected in the same directions by a perfect reflecting diffuser identically irradiated, when the thickness of the paste or the paint film is such that further increase in thickness gives no further change in the ratio.

4 Principle

4.1 Coloured and black pigments

Equal masses of a coloured pigment p_1 and of an agreed reference pigment p_2 are separately dispersed in the same mass of the same white pigment paste. The reflectivity ρ_∞ or the reflectance factor R_∞ of each dispersion is measured photometrically at a wavelength giving a minimum value of ρ_∞ or R_∞ . From the corresponding values of K/S , the relative tinting strength, K_r , of the test pigment is given by the equation

$$K_r = \frac{(K_{p1}/S)}{(K_{p2}/S)} \times 100 = \frac{K_{p1}}{K_{p2}} \times 100 \quad \dots (6)$$

where

K_{p1}/S is the K/S value corresponding to ρ_{∞} or R_{∞} of the test pigment;

K_{p2}/S is the K/S value corresponding to ρ_{∞} or R_{∞} of the agreed reference pigment.

4.2 White pigments

Equal masses of a white test pigment p_3 and of an agreed reference pigment p_4 are separately dispersed in the same mass of the same black pigment paste. The reflectivity ρ_{∞} or the reflectance factor R_{∞} of each dispersion is measured photometrically at 550 nm or using a Y-filter. From the corresponding values of K/S , the relative scattering power, S_r , of the test pigment, is given by the equation

$$S_r = \frac{(K/S_{p4})}{(K/S_{p3})} \times 100 = \frac{S_{p3}}{S_{p4}} \times 100 \quad \dots (7)$$

where

K/S_{p3} is the K/S value corresponding to ρ_{∞} or R_{∞} of the test pigment;

K/S_{p4} is the K/S value corresponding to ρ_{∞} or R_{∞} of the agreed reference pigment.

5 Materials and equipment

5.1 White pigment paste, with the following composition

- 40 parts by mass of titanium dioxide, Grade R2, complying with the requirements of ISO 591;
- 56 parts by mass of alkyd resin (see note 3 to 5.2);
- 4 parts by mass of calcium stearate.

Using a spatula, mix well so as to achieve preliminary wetting of the solids.

Then grind on the triple-roll mill (6.1) until the particle size is less than 5 μm when tested on a fineness-of-grind gauge (see ISO 1524). Store the paste in an airtight container, preferably a collapsible tube with a screw cap.

5.2 Black pigment paste (see note 1), prepared as follows.

5.2.1 Mix 18,7 parts by mass of carbon black pigment (see note 2) with 81,3 parts by mass of alkyd resin (see note 3) by use of a spatula. Pass the mixture six times through the triple-roll mill (6.1) to achieve a uniform fine dispersion.

5.2.2 Mix 3,25 g of the intermediate paste prepared as described in 5.2.1 with 91,64 g of alkyd resin (see note 3) and 5,11 g of fumed silica (see note 4); pass the mixture once through the triple-roll mill.

NOTES

1 The black pigment paste is available commercially. The mixture specified in 5.2.2 is suitable for testing at a pigment volume concentration of 17 %.

2 Carbon black pigment of the lampblack type complying with the following requirements :

		Test method
nigrometer index value	102	
volatile matter content	about 1 %	ISO 787/2
toluene extractable matter	0,15 % max.	ISO 6209
specific surface area (BET)	20 m ² /g	ISO 4652
pH value	7	ISO 787/9

3 Alkyd resin, based on a mixture of 63 % (*m/m*) linseed oil and 23 % (*m/m*) phthalic anhydride, and complying with the following requirements :

		Test method
acid value	15 mg KOH/g max.	ISO 3682
viscosity (solvent free)	7 to 10 Pa.s	ISO 3219
hydroxyl content	about 40 mg KOH/g	ISO 4629

This alkyd resin is commercially available.

4 Fumed silica complying with the following requirements :

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

6 Apparatus

6.1 Triple-roll mill.

6.2 Automatic muller, with ground glass plates, preferably water cooled (see the note), of diameter 180 to 250 mm to which a variable but known force of up to about 1 kN may be applied. The driven glass plate should have a speed of rotation of between 70 and 120 r/min and the apparatus should have an arrangement for pre-setting the number of revolutions in multiples of 25.

Pre-condition new muller plates by milling a pigment in a suitable binder (system) for 1 000 revolutions with a load applied to the plates. Remove the paste and discard.

Before use, check the surface of both plates for freedom from score marks, freedom from polished areas and for an even opaque appearance.

NOTE — If the automatic muller does not have water-cooled plates, take care that the temperature during the grinding operation does not rise by more than 10 °C.

6.3 Paste film holders, for example a suitable dish, for each test dispersion to support a paste film of about 250 μm thickness; it may have a spacing ring.

NOTE — Alternatives are to prepare a drawdown that is sufficiently thick to be completely opaque and to cover this with a template so that

an area corresponding to the sample port of the photometer is exposed, or to prepare an opaque drawdown on a glass plate and measure through the glass.

6.4 Spectrophotometer capable of measuring at wavelengths between 400 and 700 nm or a **tristimulus colorimeter** with illuminant D₆₅.

If a tristimulus colorimeter is used, appropriate filters are required for the coloured pigments under test, and a CIE Y-filter for the testing of white pigments.

NOTE — For certain purposes, it is sometimes informative to compare the scattering power of white pigments at other wavelengths; by agreement between the interested parties the tristimulus values X or Z or the spectral reflectance or the reflectance factor at an agreed wavelength may be measured and used for the calculation of the relative scattering power.

7 Sampling

Take a representative sample of the product to be tested as described in ISO 842.

8 Procedure

8.1 Determination of relative tinting strength

8.1.1 Test pigment dispersion

Weigh $3 \pm 0,01$ g of the white pigment paste (5.1) and $0,12$ g of the test sample. Place the white pigment paste in the centre of the lower plate of the automatic muller (6.2). Sprinkle the test sample on to the white pigment paste and mix together, using the minimum of effort with the aid of a spatula. Distribute the paste at several points at a distance of about 35 mm from the centre of the lower plate or spread within a ring with an internal diameter of 40 mm and an external diameter of 100 mm (see note 1).

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 four stages of 25 revolutions under a force of $1,0 \pm 0,2$ kN (see note 2). 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.

NOTES

- 1 It is advisable to lay a paper ring of the requisite shape as a pattern beneath the glass plate.
- 2 A different grinding force may be applied by agreement between the interested parties, but should be stated in the test report.

8.1.2 Reference pigment dispersion

With the agreed reference pigment, repeat the dispersion procedure described in 8.1.1, using the same mass of the agreed reference pigment and the same mass of white pigment paste (5.1).

NOTE — A different mass of the coloured pigment or another method of dispersion (for example see ISO 787/16 clause 8) may be used by agreement between the interested parties, but should be stated in the test report.

8.1.3 Preparation of test films

Place the test pigment dispersion (8.1.1) and the reference pigment dispersion (8.1.2) in the paste film holders (6.3), ensuring that the exposed surface is uniform and level.

8.1.4 Measurement of ρ_{∞} or R_{∞}

Measure ρ_{∞} or R_{∞} of each film with the photometer (see 6.4). Both measuring geometries including and excluding gloss are suitable.

If a spectrophotometer is used, vary the wavelength of light entering the photometer between 400 and 700 nm until a minimum value of ρ_{∞} or R_{∞} is obtained (at the wavelength of maximum absorption) and record the value of ρ_{∞} or R_{∞} at that wavelength for each film.

If a tristimulus colorimeter is used, select a filter that restricts the measurements to wavelengths close to that of maximum absorption. Note the reading given by the colorimeter and divide it by 100 to obtain the value of ρ_{∞} or R_{∞} .

8.2 Determination of relative scattering power

NOTE — According to the procedure described in 8.2.1 to 8.2.4 a pigment volume concentration of 17 % is chosen. This concentration is preferred because in practice the white pigment volume concentration of many air-drying paints is between 15 and 20 %.

8.2.1 Test pigment dispersion

Take a quantity of the white test sample, such that

$$m = 0,478 \rho_m \quad \dots (8)$$

where

m is the mass, in grams, of the white pigment sample;

ρ_m is the density, in grams per millilitre, of the white pigment sample, determined according to ISO 787/10 or ISO 787/23;

0,478 is a conversion factor, expressed in millilitres.

NOTE — The value of this factor is chosen to give a pigment volume concentration (p.v.c.) of 17 % with titanium dioxide pigments.

Weigh $2,5 \pm 0,01$ g of the black pigment paste (5.2) and the calculated mass of the test sample. Place the black pigment paste in the centre of the lower plate of the automatic muller (6.2). Sprinkle the test sample on to the black pigment paste and mix together, using the minimum of effort with the aid of a spatula. Distribute the paste at several points at a distance of about 35 mm from the centre of the lower plate or spread within a ring with an internal diameter of 40 mm and on an external diameter of 100 mm (see note 1).

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 four stages of 25 revolutions under a force of $1,0 \pm 0,2$ kN (see note 2). 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.

NOTES

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

2 A different grinding force may be applied by agreement between the interested parties, but should be stated in the test report.

8.2.2 Reference pigment dispersion

With the agreed reference white pigment, repeat the dispersion procedure described in 8.2.1, using the same mass m of the agreed reference white pigment as in 8.2.1 calculated for the white pigment under test, and the same mass of black pigment paste (5.2).

8.2.3 Preparation of test films

Place the test pigment dispersion (8.2.1) and the reference pigment dispersion (8.2.2) in the paste film holders (6.3), ensuring that the exposed surface is uniform and level.

8.2.4 Measurement of Q_{∞} or R_{∞}

Measure Q_{∞} or R_{∞} of each film either with a spectrophotometer using a wavelength of 550 nm or with a tristimulus colorimeter using a Y-filter. Both measuring geometries including and excluding gloss are suitable.

If a tristimulus colorimeter is used, note the reading and divide it by 100 to obtain the value of Q_{∞} or R_{∞} .

9 Expression of results

From the measured values of Q_{∞} or R_{∞} , obtain the corresponding values of K/S as given in annex B. If the measured value of Q_{∞} or R_{∞} includes gloss, subtract 0,04 before using the table in annex B. Calculate the relative tinting strength or the relative scattering power of the test sample from equation (6) or equation (7) respectively, expressed as a percentage of that of the agreed reference pigment. (See 4.1 or 4.2.)

10 Test report

The test report shall contain at least the following information :

For all pigments

- a) the type and identification of the product tested and of the agreed reference pigment;
- b) a reference to this International Standard (ISO 787/24);
- c) the measuring geometry (including or excluding gloss — see 8.1.4 and 8.2.4);
- d) the number of revolutions of the muller if different from 100 and the grinding force if different from 1 kN (see 8.1.1 and 8.2.1);
- e) any deviation, by agreement or otherwise, from the test method described;
- f) the date of the test.

For coloured and black pigments only

- g) the relative tinting strength as calculated.

For white pigments only

- h) the density values used for the calculation of the mass of pigments in the dispersions;
- j) the relative scattering power as calculated.