International Standard 787/16

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

General methods of test for pigments and extenders — Part 16: Determination of relative tinting strength (or equivalent colouring value) and colour on reduction of coloured pigments — Visual comparison method

Méthodes générales d'essai des pigments et matières de charge — Partie 16: Détermination du pouvoir colorant relatif (ou valeur de coloration équivalente) et de la couleur dégradée des pigments colorés — Méthode de comparaison visuelle

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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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 787/16 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 787/XVI-1973), clauses 0, 1, 3, 4, 5, 10 of which have been technically revised.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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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 modifications 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:

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
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 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 16 : Determination of relative tinting strength (or equivalent colouring value) and colour on reduction of coloured pigments — Visual comparison method

0 Introduction

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

This revision of ISO 787/16 has been carried out to align the presentation and procedures with those given in ISO 787/24, which describes a photometric method for comparing relative tinting strength and colour on reduction of coloured pigments. The title has been amended to differentiate between this part of ISO 787 and ISO 787/24.

The degree of development of tinting strength of a coloured pigment is dependent on the amount of work done in the preparation of the dispersion, so that in determining the relative tinting strengths of two coloured pigments it is necessary for the comparison to be carried out at the level of maximum development. In this method, which uses an automatic muller, the development of tinting strength is influenced by the force applied, the number of revolutions, the binder, the volume of the mix, and the rheology of the mix. The preliminary test described in 8.2 is used to establish the conditions under which a practical maximum of tinting strength may be obtained on the automatic muller. When these conditions are known for a particular pigment, the preliminary test is unnecessary and the procedure described in 8.3 to 8.5 is followed directly.

The complete test procedure consists of four parts:

— determination of the conditions for the preparation of the dispersion of the coloured pigment, and determination of the ratio of coloured pigment to white pigment (see 8.2);

— preparation of the dispersion of the coloured pigment (see 8.3);

— mixing of the dispersions of coloured pigment and white pigment (see 8.4);

— comparison of the colour on reduction of the two mixtures, one from the test sample and the other from the agreed reference pigment (see 8.5).

The method described is intended as a referee method. It is realized that other binders and white pigments may be used for control purposes in laboratories or by agreement between the interested parties.

For any particular application, the method of test described in this International Standard needs to be completed by the following supplementary information. This information should be derived, in part or totally, from an (inter)national standard or other document related to the product under test or, if appropriate, should be agreed between the interested parties.

a) The binder that should be used (see 5.1).

b) The volume (which should be about 2 ml) of the mix of pigment and binder.

c) The ratio of pigment to binder.

d) The ratio of coloured pigment to white pigment.

e) The force (which should be the maximum available) that should be applied to the upper plate of the automatic muller.

f) The number of revolutions of the automatic muller to be used.

1 Scope and field of application

This part of ISO 787 describes a general method of test for comparing the tinting strength and colour on reduction of two similar coloured pigments, the results being expressed either as "relative tinting strength" or as "equivalent colouring value".

ISO 787/24 describes a general method of test for determining the relative tinting strength of coloured pigments using a photometric method.

NOTES

1 When this general method is applicable to a given pigment, only a cross-reference to it 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 this general method is not applicable to a particular material should a different method for determination of relative tinting strength and colour on reduction be specified.

2 This method should not be used for those yellow pigments for which it is difficult to evaluate the tinting strength with the aid of a white pigment paste. In this case, it is common practice to use a blue pigment paste and to compare the strength and undertone of the resulting green pastes. The choice of the blue and white pigments for the blue pigment paste and its composition should be the subject of an agreement between the interested parties.

2 References

ISO 591, *Titanium dioxide pigments for paints.*

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 at defined shear rate.*

ISO 3262, *Extenders for paints.*

ISO 3668, *Paints and varnishes — Visual comparison of the colour of paints.*

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

ISO 4629, *Paint media — Determination of hydroxyl value — Titrimetric method.*[1)]

3 Definitions

3.1 white pigment paste: A dispersion of a white pigment in a binder.

3.2 reduction paste; reduced paste: A paste resulting from mixing a dispersion of a coloured pigment in a binder with a white pigment paste.

3.3 colour on reduction: The colour of a pigment when it has been incorporated in a white pigment paste (3.2).

3.4 reduction ratio: The proportion, by mass, of a coloured pigment to a white pigment in a reduction paste.

4 Principle

A dispersion of the coloured test sample, prepared under defined conditions on an automatic muller, is mixed in a known ratio with a white pigment paste. The strength and undertone of the resulting reduction paste are compared with those of a similar paste made under the same conditions from the agreed reference pigment and the same white pigment paste.

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

5 Materials

5.1 Binder

The binder shall be agreed between the interested parties. The choice of binder should be made with regard to the field of application of the pigments being tested. For example, the following binders are suggested:

NOTE — The proposed binders are available commercially.

5.1.1 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

5.1.2 Urethane-modified linseed oil, complying with the following requirements:

		Test method
linseed oil content	approximately 80 %	
acid value	nil	ISO 3682
free isocyanate groups	nil	
free hydroxyl groups	0,8 to 1,2 %	
viscosity at 20 °C	15 to 18 Pa·s	ISO 3219

5.2 White pigment paste

The composition of the white pigment paste shall be agreed between the interested parties. The choice of the white pigment paste shall be made with regard to the nature of the pigment being tested and the binder in the paste shall be compatible with the binder to be used in the coloured pigment dispersion (see 8.2.1). Unless otherwise specified, one of the following binders shall be used.

NOTE — It is strongly recommended that the same binder should be used for the white pigment paste and for the coloured dispersion as this will minimize the likelihood of flocculation and similar effects. See the note in 5.1.

5.2.1 Paste based on alkyd resin, 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 (5.1.1);

— 4 parts by mass of calcium stearate.

Using a spatula, mix well so as to achieve preliminary wetting of the solids. Then grind on a triple-roll mill until the particle size is less than 5 µm when tested on a fineness-of-grind gauge (see ISO 1524). Store in airtight containers, preferably collapsible tubes with screw caps.

5.2.2 Paste based on urethane-modified linseed oil, with the following composition:

— 40 parts by mass of titanium dioxide, Grade R2, complying with the requirements of ISO 591;

— 50 parts by mass of urethane-modified linseed oil (5.1.2);

— 7 parts by mass of calcium stearate;

— 3 parts by mass of synthetic silica (see ISO 3262).

Using a spatula, mix well so as to achieve preliminary wetting of the solids. Then grind on a triple-roll mill until the particle size is less than 5 µm when tested on a fineness-of-grind gauge (see ISO 1524). Store in airtight containers, preferably collapsible tubes with screw caps.

6 Apparatus

6.1 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.

6.2 Palette knife or **spatula,** of stainless steel or plastics material.

6.3 Panels, made of glass or other transparent, colourless, non-absorbent material.

6.4 Film of plastics material, transparent and colourless.

6.5 Film applicator, suitable for applying two or three films side by side of wet thickness 50 to 100 µm.

7 Sampling

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

8 Procedure

8.1 Milling concentrations

8.1.1 The appropriate 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 initially be allocated to one of three groups:

a) pigments having a low binder demand — average milling concentration 66,7 % (m/m) of pigment;

b) pigments having an intermediate binder demand — average milling concentration 40 % (m/m) of pigment;

c) pigments having a high binder demand — average milling concentration 25 % (m/m) of pigment.

8.1.2 In order to give approximately 2 ml of mix in each case, the quantities to be used in the test for the three groups defined in 8.1.1 shall be the following:

a) 3,0 g of pigment and 1,5 g of binder;

b) 1,0 g of pigment and 1,5 g of binder;

c) 0,5 g of pigment and 1,5 g of binder.

NOTES

1 If the mix chosen is found to be unsuitably stiff or fluid for use on the muller, one of the other ratios should be used as appropriate.

2 If the diameter of the muller plates is near the maximum of the range specified in 6.1, it may be necessary to increase the amounts specified in order to reduce wear on the plates.

8.2 Preliminary test for establishing dispersion conditions

8.2.1 Weigh approximately 1,5 g of the binder (5.1) and the appropriate quantity of the agreed reference pigment. Place the binder in the centre of the lower plate of the automatic muller. Sprinkle the pigment on to the binder and mix together, using the minimum of effort with the aid of the spatula (6.2). 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. 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 maximum applied force. 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 a total of 200 revolutions, remove a small portion (about one-quarter of the total volume) of the collected paste, store it in a suitable receptacle and continue grinding the main bulk. Remove similar small portions after totals of 300 and 400 revolutions, store them in suitable receptacles and then clean the muller plates and spatula.

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

8.2.2 Place on the lower plate of the muller

— 3 ± 0,01 g of the white pigment paste (5.2);

— an amount of the coloured pigment paste which has been ground for 200 revolutions and which contains about 0,12 g of the coloured pigment (see the note).

Mix the two pastes with the spatula as homogeneously as possible, without any grinding action, and spread the mixture on the lower plate as described in 8.2.1, wiping the spatula on the upper plate as before. Close the plates and, with the minimum applied force, grind in stages of 25 revolutions of the muller. After each stage, collect the paste on the lower plate as described in 8.2.1 until four stages have been completed. Then remove the paste from the muller and set aside for later assessment. Repeat this operation using other portions of coloured paste that have been ground for 300 to 400 revolutions respectively and contain 0,12 g of the coloured pigment.

NOTE — The amount of coloured pigment paste which contains 0,12 g of coloured pigment will, when mixed with 3 g of white pigment paste, give a reduction ratio of 1 : 10. This ratio should be modified to, for example, 1 : 5 or 1 : 20 (to suit weak pigments or strong pigments respectively) in order to produce a colour on reduction of intensity that is suitable for assessing the strength and undertone of the reduction pastes.

8.2.3 Apply each of these reduction pastes in order, side by side, with touching edges on a glass panel (6.3) or transparent film of plastics material (6.4).

Compare visually the strength of colour of each of the pastes. Assess the paste which has developed the maximum strength of colour and record the number of revolutions of the muller necessary to obtain that paste. (If more than one paste has developed the same maximum strength of colour, record the lowest number of revolutions.) Use this number of revolutions for the test itself.

8.3 Preparation of dispersion of coloured pigment

8.3.1 On the basis of the information obtained from 8.2 decide:

a) the quantities of pigment and binder to be used in preparing the dispersion of the coloured agreed reference pigment;

b) the number of revolutions to be used in preparing the coloured agreed reference pigment dispersion and the force used;

c) the reduction ratio to be used for mixing the coloured pigment dispersion with the white pigment paste.

8.3.2 Applying these decisions, prepare a dispersion on the automatic muller of the agreed reference pigment as described in 8.2, but carrying out the grinding in stages of 50 revolutions to the full number of revolutions previously decided without removing any of the paste, but gathering and spreading the paste after each stage. When the grinding has been completed, collect the paste and store it in a suitable receptacle. Clean the muller and spatula, and repeat the operation with the same quantities of the test sample and of the binder and using the same procedure on the same muller. Collect the coloured pigment paste from this sample and store it in a suitable receptacle. Clean the muller and spatula.

8.4 Preparation of the reduction paste

8.4.1 Place on the lower muller plate:

— 3 ± 0,01 g of the white pigment paste (5.2);

— an amount of the coloured pigment paste, prepared as described in 8.3.2, containing the mass of coloured agreed reference pigment to give the chosen reduction ratio.

Mix the two pastes with the spatula as homogeneously as possible, without any grinding action, and spread the paste on the lower plate as described in 8.2.1, wiping the spatula on the upper plate. Close the plates and, with the minimum applied force, grind in stages of 25 revolutions of the muller. After each stage, collect the paste on the lower plate as described in 8.2.1, until four stages have been completed. Then remove the paste from the muller and store in a suitable receptacle for the later assessment (8.5).

8.4.2 Repeat the operation using the coloured pigment dispersion paste prepared from the test sample of the coloured pigment, thus producing the comparative reduced paste.

8.5 Comparison of colour on reduction and determination of relative tinting strength

8.5.1 Place a quantity of each of the two reduced pastes obtained as described in 8.4 side by side on a glass panel (6.3) or transparent film of plastics material (6.4) (see the note). With the film applicator (6.5) draw the pastes down to form two uniformly thick strips not less than 25 mm wide and not less than 40 mm long, with touching edges.

Both strips shall have such a film thickness that the substrate is completely hidden. Lightly rub a part of each strip with a finger. Compare the difference in depth of shade between the rubbed and non-rubbed surfaces and record if a significant difference is observed. Continue the test, examining only the non-rubbed surface. Using the procedure described in ISO 3668 and, immediately after application, compare for strength and for undertone by examining the two strips in diffuse daylight on the surface or, by agreement between the interested parties, through the glass or plastics film. Where good daylight is not available, make the comparison in artificial daylight using the procedure described in ISO 3668.

If the strengths are equal and the undertones are the same, the colours on reduction are the same, and the relative tinting strength of the sample under test is 100 % (see 9.1).

However, if the strengths are equal but the undertones are not the same, note the difference in colour on reduction and its nature.

NOTE — Flocculation of the reduction paste may be indicated if the appearance of the films, when viewed directly, differs from their appearance when viewed through the substrate. In this instance, the results of the test should be considered to be suspect.

8.5.2 If the strengths are considered to be unequal, and therefore the colours on reduction are not the same, repeat the operations of 8.4 and 8.5.1, but weighing and using a quantity of the coloured pigment dispersion of the test sample estimated to give a strength equal to that of the original mass taken of the coloured pigment dispersion of the agreed reference pigment.

If there is also a difference in undertone, note the difference and its nature.

NOTE — For example, if it is considered that the sample under test is 15 % stronger than the agreed reference pigments, then in the repeat test, the mass of the coloured pigment dispersion of the test pigment used should be 15 % less, but the mass of coloured pigment dispersion of the agreed reference pigment should be the same as that originally used.

9 Expression of results

9.1 Calculate the relative tinting strength of the test sample by the formula

$$\frac{b \times 100}{a} \text{ \% of the agreed reference pigment}$$

where a is the number of parts by mass of the test sample required to produce the same strength as b parts by mass of the agreed reference pigment.

NOTE — It is important to include the per cent sign (%), and to note that the tinting strength is relative to that of the agreed reference pigment as 100 %.

9.2 Calculate the equivalent colouring value of the test sample by the formula

$$\text{weaker or stronger } \frac{a \times 100}{b} : 100$$

NOTE — It is important to include the word "weaker" if the value is more than 100, or "stronger" if the value is less than 100.

Example A: If a = 20 parts and b = 25 parts, then the relative tinting strength of the test sample is **125 %**, and the equivalent colouring value of the test sample is **stronger 80 : 100**.

Example B: If a = 50 parts and b = 45 parts, then the relative tinting strength of the test sample is **90 %**, and the equivalent colouring value of the test sample is **weaker 111 : 100**.

10 Test report

The test report shall contain at least the following information:

a) the type and identification of the product tested;

b) a reference to this International Standard (ISO 787/16);

c) the number of revolutions used in preparing the dispersion of the coloured pigment (see 8.3.2);

d) the reduction ratio used to prepare the reduction paste (see the note to 8.2.2);

e) whether the test films were examined under natural or artificial daylight (see 8.5.1);

f) the result of the comparison of the colour on reduction, if possible with a qualitative statement concerning any difference in undertone;

g) the relative tinting strength (9.1) or the equivalent colouring value (9.2);

h) any deviation, by agreement or otherwise, from the procedure specified, including if the test was carried out by examining through the substrate (see 8.5.1);

i) the date of the test.