INTERNATIONAL STANDARD ISO 787

PARTS XIII TO XVIII

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General methods of test for pigments – Parts XIII to XVIII

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

International Standard ISO 787/XIII to XVIII (originally Draft International Standard ISO/DIS 2304) was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes,* and circulated to the Member Bodies in April 1971.

It has been approved by the Member Bodies of the following countries :

Austria
Egypt, Arab Rep. of
France
Germany
India
Israel

Italy
Netherlands
New Zealand
Poland
Romania
South Africa, Rep. of

Sweden
Switzerland
Turkey
United Kingdom

No Member Body expressed disapproval of the document.

Printed in Switzerland

The purpose of this International Standard is to establish a series of general test methods for pigments which are suitable for all or many of the individual pigments 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, with a note of any detailed modifications which might be needed in view of the special properties of the pigment in question.

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

The Committee also decided that, where two or more procedures were widely used for determining the same or a similar characteristic of a pigment, 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 I : Comparison of colour
Part II : Determination of matter volatile at 105 °C
Part III : Determination of matter soluble in water (Hot extraction method)
Part IV : Determination of acidity or alkalinity of the aqueous extract
Part V : Determination of oil absorption value
Part VI : Determination of residue on sieve (Oil method)
Part VII : Determination of residue on sieve (Water method)
Part VIII : Determination of matter soluble in water (Cold extraction method)
Part IX : Determination of pH value of an aqueous suspension
Part X : Determination of density relative to water at 4 °C
Part XI : Determination of tamped volume
Part XII : Visual comparison of hue of powered white pigment (Hollow cone method)

The present document contains the following parts :

Part XIII : Determination of water-soluble sulphates, chlorides and nitrates
Part XIV : Determination of resistivity of aqueous extract
Part XV : Comparison of resistance of coloured pigments of similar types to light from a specified light source
Part XVI : Comparison of relative tinting strength (or equivalent colouring value) and colour on reduction in linseed stand oil using the automatic muller
Part XVII : Comparison of lightening power of white pigments
Part XVIII : Determination of residue on sieve by a mechanical flushing procedure

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General methods of test for pigments – Part XIII : Determination of water-soluble sulphates, chlorides and nitrates

0 INTRODUCTION

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

1 SCOPE AND FIELD OF APPLICATION

Part XIII of this International Standard specifies a general method of test for determining the water-soluble sulphates, chlorides and nitrates of pigments.

NOTE – When this general method is applicable to a given pigment, a cross-reference to it will simply be included in the International Standard relating to that pigment, with a note of any detailed modification which may be needed in view of the special properties of the pigment in question. Only when this general method is not applicable to a particular pigment will a special method for determination of water-soluble sulphates, chlorides or nitrates be specified.

2 REAGENTS

All reagents used shall be of recognized analytical reagent quality. Distilled water, or water of equivalent purity, shall be used.

2.1 Hydrochloric acid, $d = 1{,}18$.

2.2 Silver nitrate, 0,01 N standard volumetric solution.

2.3 Ammonium chloride solution, 17,2 mg/l.

2.4 Sodium hydroxide solution, 200 g/l.

2.5 Barium chloride solution, 50 g/l.

2.6 Potassium chromate solution, 50 g/l.

2.7 Devarda's alloy, powdered.

2.8 Nessler's reagent, prepared by either method a) or method b) as follows :

a) Dissolve 5 g of potassium iodide in 3,5 ml of water. Add cold saturated mercury(II) chloride ($HgCl_2$) solution, while stirring, until a faint red precipitate is formed. Continuing to stir, add 40 ml of potassium hydroxide solution (500 g/l), dilute to 100 ml, mix well, allow to settle, decant the clear supernatant liquid and store it in the dark.

b) Dissolve 3,5 g of potassium iodide and 1,25 g of mercury(II) chloride in 80 ml of water. Add cold saturated mercury(II) chloride solution, while shaking, until a slight red precipitate remains, then add 12 g of sodium hydroxide, shake until dissolved, and finally add a little more of the saturated mercury(II) chloride solution and dilute to 100 ml with water. Shake occasionally during several days, allow to stand, and use the clear supernatant liquid for the test.

3 APPARATUS

Normal laboratory equipment and

3.1 Sintered silica crucible, porosity grade P10 or P16 (pore size index 4-16 μm).

3.2 Nessler cylinders, capacity 50 ml.

3.3 Distillation apparatus.

4 SAMPLING

The sample of pigment used for the test shall be taken in accordance with the provisions of ISO/R 842, *Sampling raw materials for paints and varnishes.*

5 DETERMINATION OF SULPHATES

5.1 Procedure

Take 50 ml of the clear aqueous extract obtained in one of the methods, as appropriate, for the determination of matter soluble in water (either the hot extraction method[1] or the cold extraction method[2]), acidify with 3 ml of the hydrochloric acid (2.1) and boil the solution vigorously, taking care to avoid loss of solution by splashing. Add the barium chloride solution (2.5), drop by drop, to the hot solution until in slight excess, and allow the solution to

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1) See Part III.

2) See Part VIII.

stand overnight. Decant the supernatant liquid through the tared silica crucible, transfer the precipitate to the crucible and wash it free from chloride, ignite it gently, then at red heat, cool it in a desiccator and weigh to the nearest 1 mg.

5.2 Expression of results

Calculate the water-soluble sulphate content expressed as SO_4, as a percentage by mass, by the formula :

$$\frac{206\, m_1}{m_0}$$

where

m_0 is the mass, in grams, of pigment used in the determination of matter soluble in water;

m_1 is the mass, in grams, of barium sulphate precipitate.

Report the result to two decimal places.

6 DETERMINATION OF CHLORIDES

6.1 Procedure

Take 50 ml of the clear aqueous extract obtained in one of the methods, as appropriate, for the determination of matter soluble in water (either the hot extraction method[1] or the cold extraction method[2] and add 1 ml of the potassium chromate solution (2.6). Titrate with the silver nitrate solution (2.2), slowly and with vigorous shaking, until a faint reddish brown colour persists.

Carry out a blank determination by adding 1 ml of potassium chromate solution to 50 ml of water and titrate with the silver nitrate solution until the colour matches that of the previous titration, making due allowance for any opalescence or turbidity.

NOTE — Alternatively the end-point of the titration may be determined by potentiometric indication.

6.2 Expression of results

Calculate the water-soluble chloride content expressed as Cl, as a percentage by mass, by the formula :

$$0{,}177\,5\,\frac{(V_1 - V_0)}{m}$$

where

V_0 is the volume, in millilitres, of 0,01 N silver nitrate solution required for the blank determination;

V_1 is the volume, in millilitres, of 0,01 N silver nitrate solution required by the test portion;

m is the mass, in grams, of pigment used in the determination of matter soluble in water.

Report the result to two decimal places.

7 DETERMINATION OF NITRATES

7.1 Procedure

Place 50 ml of the clear aqueous extract obtained in one of the methods, as appropriate, for the determination of matter soluble in water (either the hot extraction method[1] or the cold extraction method[2] in the distillation flask (3.3) and dilute to 150 ml. Add 3 g of Devarda's alloy (2.7) and 30 ml of the sodium hydroxide solution (2.4) and close the apparatus at once. Place 2 ml of the hydrochloric acid (2.1) and 30 ml of water in the receiver.

Warm the flask gently until the reaction starts and then allow the reaction to proceed gently for about half an hour. Then distil about 70 ml of liquid, the receiver being kept cool with running water.

Make up the distillate to 250 ml with water and transfer 5 ml to a Nessler cylinder (3.2). Dilute to 50 ml. Add 1 ml of Nessler's reagent (2.8) and match the colour against that of a similar standard solution prepared by adding ammonium chloride solution (2.3) from a burette.

Carry out a blank determination using 50 ml of distilled water.

7.2 Expression of results

Calculate the water-soluble nitrate content expressed as NO_3, as percentage by mass, by the formula :

$$0{,}5\,\frac{(V_1 - V_0)}{m}$$

where

V_0 is the volume, in millilitres, of ammonium chloride solution required by the blank determination;

V_1 is the volume, in millilitres, of ammonium chloride solution required by the test portion;

m is the mass, in grams, of pigment used in the determination of matter soluble in water.

Report the result to two decimal places.

8 TEST REPORT

The test report shall include the following information :

a) a reference to ISO 787, Part XIII, or to a corresponding national standard;

b) type and identification of the pigment under test;

c) any deviation, by agreement or otherwise, from the test procedure described above;

d) whether the aqueous extract for the test was obtained by the hot extraction method or the cold extraction method;

e) the result of the test as indicated by 5.2, 6.2 or 7.2;

f) the date of the test.

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[1] See Part III.

[2] See Part VIII.

General methods of test for pigments – Part XIV : Determination of resistivity of aqueous extract

0 INTRODUCTION

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

1 SCOPE AND FIELD OF APPLICATION

Part XIV of this International Standard specifies a general method of test for determining the resistivity (specific resistance) of the aqueous extract of a pigment. The method is applicable to all pigments and extenders, except pigments that are substantially soluble in water.

NOTES

1 It should be noted that the resistivity of the aqueous extract of a pigment should be considered as a property, independent of the amount of water-soluble matter. If agreed, a cold extraction method may be used. This shall be stated in the test report.

2 The standard temperature of determination should be 23 °C but a different temperature may be agreed between the parties provided that the necessary corrections are made to take account of the differences in temperature.

3 When this general method is applicable to a given pigment, a cross-reference to it will simply be included in the International Standard relating to that pigment, with a note of any detailed modification which may be needed in view of the special properties of the pigment in question. Only when this general method is not applicable to a particular pigment, will a special method for determination of resistivity of aqueous extract be specified.

2 REAGENTS

All reagents used shall be of recognized analytical reagent quality.

2.1 Conductivity water, resistivity not less than 2 500 Ω·m.

2.2 Methanol, resistivity not less than 2 500 Ω·m.

2.3 Potassium chloride, 0,02 M solution.

3 APPARATUS

3.1 Centrifuge, or ultra-centrifuge if necessary.

3.2 Filter paper, fine-textured, which should be washed with conductivity water on a filter funnel until the filtrate gives a resistivity greater than 2 000 Ω·m.

NOTE – The diameter of the filter paper depends on the bulk density of the pigment. Some organic pigments require at least a 185 mm paper for satisfactory filtering.

3.3 Cylinders, approximately 35 mm wide by 125 mm deep, or other containers suitable for use with the conductivity cell to be employed.

3.4 Thermometer, graduated in 0,2 °C intervals.

3.5 Conductivity bridge[1].

3.6 Conductivity cell,[1] having a cell constant, *K*, of approximately 1.

4 SAMPLING

The sample of pigment used for the test shall be taken in accordance with the provisions of ISO/R 842, *Sampling raw materials for paints and varnishes.*

5 DETERMINATION OF CELL CONSTANT

5.1 Prepare a working standard solution of potassium chloride by diluting the potassium chloride solution (2.3) with conductivity water to a known concentration (see Notes 1 and 2). Measure the resistance *R* of this solution using the conductivity cell (3.6) at 23 °C (or at an alternative agreed temperature with appropriate corrections) as described in 6.2.2.

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[1] Any commercially produced conductivity bridge and conductivity cell are likely to be satisfactory.

5.2 Calculate the cell constant, K, as follows :

$$K = \frac{R}{\rho}$$

where

R is the measured resistance, in ohms;

ρ is the resistivity at 23 °C of KCl solution of the concentration used, in ohm metres (for a 0,002 M solution this is 34,4 Ω·m, see Figure).

NOTES

1 If a potassium chloride solution of different known concentration is used, the appropriate value of ρ should be deduced from the Figure for use in the calculation of the cell constant.

2 In general the cell constant is not greatly affected by variations in the strength of the potassium chloride solution, but for greatest accuracy a concentration of the potassium chloride solution shall be used which has a resistivity similar to that of the solution being tested, and measurements shall be made at values that utilise the middle third of the scale.

6 PROCEDURE

6.1 Test for water-wettability of the pigment

Test a small amount of the pigment with boiling distilled water to see if it is water-wettable. Material which does not wet well with water is probably hydrophobic and shall be treated as described in 6.3. If the sample wets easily, proceed as described in 6.2.

6.2 Hydrophilic pigments

6.2.1 Add 20 ± 0,01 g of the pigment to 180 g of boiling conductivity water in a tared beaker of suitable capacity with a stirring rod.

NOTE — A 20 g sample is usually sufficient for pigments easily wetted with water. Usually a 250 ml beaker is sufficient for white pigments. Some white pigments, however, because of tendency to foam and crawl, can be handled better in a 400 ml beaker. A 20 g sample of an organic pigment usually requires a 600 ml beaker to allow adequate room for foaming when boiled.

Boil slowly for 5 min with occasional stirring. Cool to a temperature of about 60 °C and add water to bring the net mass back to 200 g. Stir thoroughly. Filter directly through a fine-textured filter paper, or separate the solids using a centrifuge or ultra-centrifuge and clean dry tubes, or tubes washed with some of the slurry, followed by decanting the supernatant liquid through a filter. In either case discard the first 10 ml of filtrate.

6.2.2 Cool the filtrate to a temperature of about 20 °C. Rinse the cylinder (3.3) and the conductivity cell (3.6) first with conductivity water and then with the filtrate. Fill the cylinder with the filtrate and place the conductivity cell in it. Move the cell up and down to remove all air bubbles. Adjust the temperature slowly to 23 °C and with the cell submerged so that the vent is about 10 mm below the surface of the liquid, and upright in the centre of the cylinder, make at least five measurements of the resistance at a temperature of 23 ± 0,5 °C, using the conductivity bridge (3.5) with the multiplier set to give a reading near the centre of the scale, following the instructions supplied with the instrument to obtain a balance.

6.3 Hydrophobic pigments

A modification of the procedure given in 6.2 is necessary for organic pigments that are not easily wetted with water.

Wet 20 ± 0,01 g of pigment with as much of the methanol (2.2) (4 to 16 g) as is required to produce a smooth wet paste. Dilute with boiling conductivity water in a tared 1 000 ml beaker with a stirring rod to bring the total mass to 200 mg. Then proceed as outlined in 6.2.2.

6.4 Repeat the whole procedure on a further aqueous extract of the pigment.

7 EXPRESSION OF RESULTS

Calculate the resistivity ρ_t, in ohm metres, of the aqueous extract of the pigment at the agreed temperature t °C, by the formula :

$$\rho_t = \frac{\overline{R}_t}{K}$$

where

$\overline{R}_t$ is the mean of all the measured values of resistance, in ohms,

K is the cell constant, determined in accordance with 5.2.

Express the result to the nearest 1 % of the value obtained.

8 TEST REPORT

The test report shall include the following information :

a) a reference to ISO 787, Part XIV, or to a corresponding national standard;

b) type and identification of the pigment under test;

c) any deviation, by agreement or otherwise, from the test procedure described above;

d) whether the pigment was treated as a hydrophilic pigment (6.2) or a hydrophobic pigment (6.3);

e) the result of the test as indicated in section 7;

f) the date of the test.

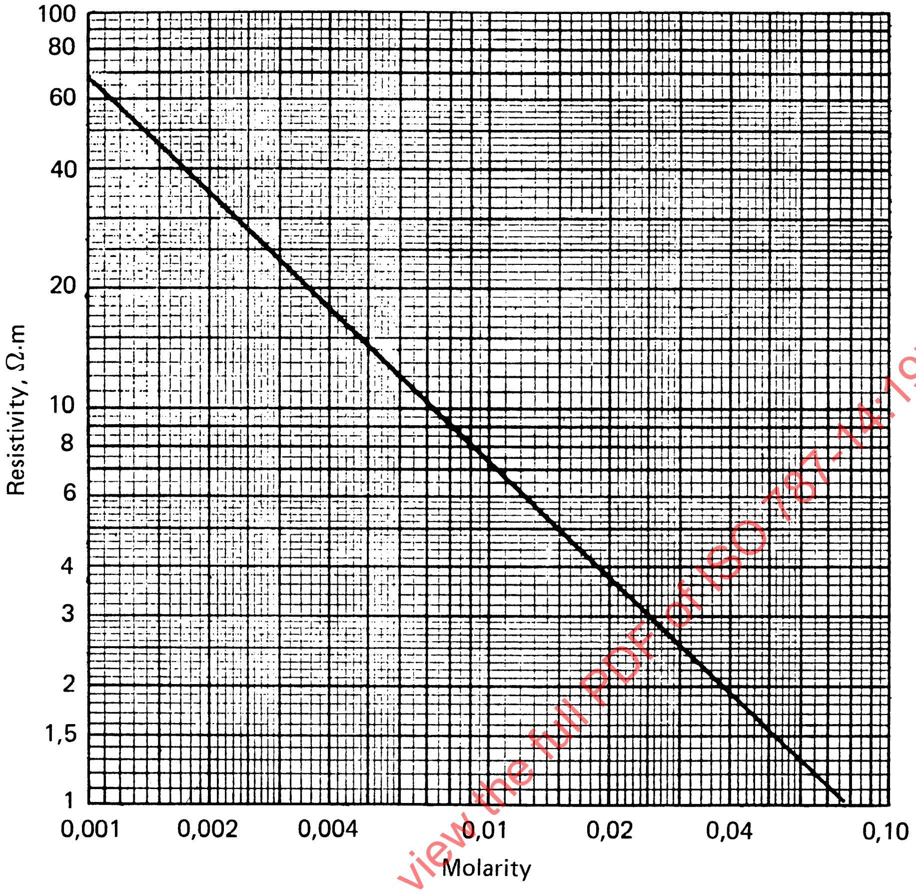

FIGURE — **Resistivity of potassium chloride at 23 °C**

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General methods of test for pigments – Part XV : Comparison of resistance of coloured pigments of similar types to light from a specified light source

0 INTRODUCTION

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

1 SCOPE AND FIELD OF APPLICATION

Part XV of this International Standard specifies a general method of test for comparing the resistance to light of two samples of similar types of coloured pigment.

NOTE – When this general method is applicable to a given pigment, a cross-reference to it will simply be included in the International Standard relating to that pigment, with a note of any detailed modification which may be needed in view of the special properties of the pigment in question. Only when this general method is not applicable to a particular pigment will a special method for comparison of resistance to light be specified.

2 PRINCIPLE

The terms "resistance to light" and "lightfastness" describe the resistance of a material to change in its appearance as a result of exposure to light. The magnitude of the change, if any, is influenced by the quantity and quality of the light to which the material is exposed, and by the nature and composition of the material itself. Two compositions each consisting of identical components, but in different proportions, may not have the same resistance to light as each other. Also, two compositions each consisting of the same proportions of similar, but not identical, components may not have the same resistance to light.

Experience of this situation is the basis for the comparison of lightfastness of two different samples used as a component in a particular composition, for example, a coloured pigment. Each sample is incorporated in the same proportion in otherwise identical compositions and the latter, in a suitable form, are examined for any difference in the amount of change of appearance after exposure to the same quantity and quality of light. In order to comply with these exposure conditions, it is necessary for the compositions to be exposed side by side at the same time to the same light source for the same period of time, since it is well known that the quantity and quality of daylight varies from day to day, and that the light from artificial daylight lamps varies with age.

The extent to which the change on exposure is allowed to proceed before comparison is made, may be of importance. It is unrealistic to assess the exposures when the change is only equivalent to the first perceptible change, but it is also unhelpful to wait until the amount of change is large. It is recommended that the comparison of change of appearance should be made when the amount of change is equal to Grade 3 of the grey scale, as described in Part 2 of ISO/R 105/I, *Tests for colour fastness of textiles – First series.* It is emphasised that the test described below is designed to compare the lightfastness of test specimens which are exposed to the same light source at the same time.

3 APPARATUS

3.1 Substrate. Unless otherwise agreed, cardboard pieces of suitable size for the applicator used, of a rigid quality and with a white, high gloss, lightfast, coated, non-absorbent surface suitable for the application of paint.

3.2 Film applicator or other device, suitable for applying two films side by side of wet thickness 50 to 100 μm.

3.3 Cover sheet, of aluminium foil or other suitable opaque material.

3.4 Geometric grey scale¹⁾ complying with Part 2 of ISO/R 105/I.

3.5 Source of light. Natural daylight is the preferred source but artificial sources of light may be used by agreement between the parties.

A mercury vapour lamp is not suitable for this test.

4 SAMPLING

The sample of pigment used for the test shall be taken in accordance with the provisions of ISO/R 842, *Sampling raw materials for paints and varnishes.*

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1) Obtainable from most national standards institutions.

5 PROCEDURE

5.1 Preparation of test specimen

Prepare dispersions of the test sample and the reference sample in an agreed medium. By means of the film applicator (3.2) or other device, apply to the substrate (3.1) continuous films of both dispersions so that each is at least 25 mm wide (see Note). Allow to dry in a horizontal position in diffuse daylight for 24 h at normal room temperature. Alternatively, stove under the specified conditions. Cut a suitably sized test specimen to fit the exposure frame, if used, so that the dividing line between the two dispersion films is central.

NOTE — If it is intended to carry out instrumental measurements, care shall be taken that the test specimen is of sufficient size.

5.2 Exposure of test specimen

5.2.1 Fasten a cover sheet across the middle one-third of the test specimen, clipping down the edges to prevent buckling, but in such a way that the cover can be removed for examination of the films and replaced in the same position.

5.2.2 For exposure to daylight, the test specimen should be mounted behind window glass, parallel to it and at a distance of about 50 mm from it, protected from the weather, but with free circulation of air. It is desirable that the window glass should be facing south in the northern hemisphere, and north in the southern hemisphere, and inclined to the horizontal at an angle equal to the latitude of the place of exposure.

5.2.3 Expose the test specimen to the source of light, and at suitable intervals of time examine it by raising the cover to determine if there is any change between the exposed and unexposed portions, replacing the cover in the same position. When the contrast between the exposed and unexposed portions of the reference sample is equal to that illustrated by Grade 4 of the geometric grey scale (3.4), record the degree of contrast of the film of the test sample, and replace the cover.

5.2.4 Apply a further cover sheet so that two-thirds of the test specimen is covered and continue the exposure until the fully exposed and the central unexposed portions of the film of the reference sample show a contrast equal to Grade 3 of the geometric grey scale. Assess the amount of contrast or change of appearance of the film of the test sample in relation to that of the film of the reference sample.

6 TEST REPORT

The test report shall include the following information :

a) a reference to ISO 787, Part XV, or to a corresponding national standard;

b) type and identification of the pigment under test;

c) any deviation, by agreement or otherwise, from the test procedure described above;

d) the medium used, and details of the dispersion used;

e) whether, at the various stages of the test, the change in appearance of the film of the test sample is greater than, equal to, or less than, the change in appearance of the film of the agreed sample;

f) details of light source and whether instrumental or visual methods of assessment were used;

g) the date of the test.

General methods of test for pigments — Part XVI : Comparison of relative tinting strength (or equivalent colouring value) and colour on reduction in linseed stand oil using the automatic muller

0 INTRODUCTION

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

1 SCOPE AND FIELD OF APPLICATION

Part XVI of this International Standard specifies a general method of test for comparing the tinting strength and colour on reduction of two similar coloured pigments in linseed stand oil, the results being expressed either as "relative tinting strength" or as "equivalent colouring value".

NOTE — When this general method is applicable to a given pigment, a cross-reference to it will simply be included in the International Standard relating to that pigment, with a note of any detailed modification which may be needed in view of the special properties of the pigment in question. Only when this general method is not applicable to a particular pigment, will a special method for comparison of tinting strength and colour on reduction be specified.

2 PRINCIPLE

2.1 A dispersion of the coloured pigment under test, prepared under known conditions on an automatic muller, is mixed in a known ratio with a dispersion of white pigment. The intensity of colour and hue of the resulting paste is compared with that of a similar paste made under the same conditions from the agreed sample of coloured pigment and the same dispersion of white pigment.

2.2 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, and 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 medium, the volume of the mix, and the rheology of the mix. By following the procedure described in 6.1, the conditions are determined under which a practical maximum of tinting strength may be obtained on the automatic muller. When these conditions are known for a particular pigment, there is no need to carry out the procedure described in 6.1.

2.3 Of the factors listed in 2.2, the following can be specified without difficulty :

a) the force applied, which shall be the maximum available;

b) the medium to be used, which is linseed stand oil;

c) the volume of the mix of pigment and medium, which shall be about 2 ml.

The remaining factors to be decided, which shall be chosen according to the level of tinting strength of the coloured pigment under test, are the following :

d) the ratio of pigment to medium;

e) the number of revolutions to be used;

f) the ratio of coloured pigment to white pigment.

2.4 The appropriate ratio of pigment to medium, by mass, depends not only on the oil absorption of the pigment but also on the viscosity of the mix during the mulling operation. As a first step all pigments can initially be allocated to one of three groups :

a) pigments of low medium requirement — average milling concentration 66,7 % of pigment by mass;

b) pigments of intermediate medium requirement — average milling concentration 40 % of pigment by mass;

c) pigments of high medium requirement — average milling concentration 25 % of pigment by mass.

2.5 The quantities to be used in the test for the three groups defined in 2.4 shall be the following, in order to give approximately 2 ml of mix in each case :

a) 3,0 g of pigment and 1,5 g of linseed stand oil;

b) 1,0 g of pigment and 1,5 g of linseed stand oil;

c) 0,5 g of pigment and 1,5 g of linseed stand oil.

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 at the top end of the range specified in 4.1, it may be necessary to increase the amounts specified in order to reduce wear on the plates.

2.6 The complete test procedure consists of four parts :

a) 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 6.1);

b) preparation of the dispersion of the coloured pigment (see 6.2);

c) mixing of the dispersions of coloured pigment and white pigment (see 6.3);

d) comparison of the intensity of colour and hue of the two mixtures, one from the sample under test and the other from the reference sample (see 6.4).

2.7 The method described is intended as a reference method and the medium for preparing the dispersions is therefore specified, although it is realised that other media and white pigments may be used for control purposes in laboratories or by agreement between the parties to a test.

NOTE – Both the standard medium (linseed stand oil as defined in 3.1) and the standard white paste (titanium dioxide in urethane linseed oil, as defined in 3.2) are available commercially.

3 REAGENTS

3.1 Linseed stand oil, complying with the requirements of ISO/R 276, *Linseed stand oils,* of acid value 8 to 12 mg KOH per gram, and viscosity at 20 °C of 4,5 to 5,5 N·s/m².

3.2 White pigment paste, with the following composition :

- 40 parts by mass of titanium dioxide, Type R, complying with the requirements of ISO/R 591, *Titanium dioxide for paints*;
- 50 parts by mass of urethane modified linseed oil having the following characteristics :
  - linseed oil content : approximately 80 %
  - acid value : nil
  - free isocyanate groups : nil
  - free hydroxyl groups : 0,8 to 1,2 %
  - viscosity at 20 °C : 15 to 18 N·s/m²
- 7 parts by mass of calcium stearate;
- 3 parts by mass of synthetic silica.

Mix well so as to achieve careful 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. Store in airtight containers, preferably tubes with screw caps.

4 APPARATUS

4.1 Automatic muller, with ground glass plates, preferably water-cooled[1], of diameter 180 to 250 mm to which a variable but known force of up to about 1 000 N may be applied. The driven glass plate shall have a speed of rotation of between 70 and 120 rev/min and the apparatus shall have an arrangement for pre-setting the number of revolutions in multiples of 25.

4.2 Palette knife or **spatula** of steel or plastics material.

4.3 Glass slides, or other transparent, colourless, non-absorbent substance.

4.4 Film of plastics material, transparent and colourless.

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

4.6 Balance, accurate to ± 0,001 g.

5 SAMPLING

The sample of pigment used for the test shall be taken in accordance with the provisions of ISO/R 842, *Sampling raw materials for paints and varnishes.*

6 PROCEDURE

6.1 Determination of conditions for preparing the dispersion of the coloured pigments

6.1.1 Weigh the appropriate quantity of medium as specified in 2.5 in the chosen group and transfer it to the lower plate of the muller (4.1); weigh the corresponding quantity of the reference sample of the coloured pigment and sprinkle it carefully on the medium. Mix the pigment with the medium by means of the spatula (4.2), but do not grind. Spread the mixture in an approximately circular patch, of diameter about 100 mm, in the centre of the lower plate and clean the spatula as much as possible by wiping it on the upper plate of the muller. Close the plates and carry out the grinding in stages of 50 revolutions; after each stage collect the paste with the spatula from both plates and spread it out in an approximately circular patch on the centre of 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, set this aside and continue grinding the main bulk. Remove similar small portions after totals of 300 and 400 revolutions, set these aside also and then clean the muller plates and spatula.

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[1] If the automatic muller does not have water-cooled plates, care shall be taken that temperature variation does not occur during the grinding operation.

6.1.2 Place on the lower muller plate 3 ± 0,01 g of white pigment paste and an amount of the coloured pigment paste which has been ground for 200 revolutions which contains 0,12 g of coloured pigment (see 6.1.4) and mix the two pastes with the spatula as homogeneously as possible, without grinding. Spread the mixture in an approximately circular patch in the centre of the lower plate, wipe the spatula on the upper plate, close the plates and grind for four stages of 25 revolutions each with the minimum applied force; after each stage collect the paste in the centre of the lower plate as previously described. Then remove the paste from the muller and set aside for later assessment. Repeat this operation with the same mass of each of the portions of coloured paste which have been ground for 300 and 400 revolutions respectively.

6.1.3 Apply each of these reduction pastes in order, side by side, with touching edges on a sheet of glass (4.3), or transparent film of plastics material (4.4), so that the intensity of colour can be judged visually. Record the minimum number of revolutions necessary to obtain the sample of coloured pigment paste which produces the maximum intensity of colour and use this number of revolutions for the test itself.

6.1.4 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 which is suitable for assessing the intensity and hue of the reduction pastes.

6.2 Preparation of dispersion of coloured test pigments

6.2.1 On the basis of the information obtained from 6.1, decide :

a) the quantities of pigment and medium to be used in preparing the coloured test pigment dispersion;

b) the number of revolutions to be used in preparing the coloured test pigment dispersion;

c) the ratio to be used for preparing the reduction with the white pigment paste.

6.2.2 Applying these decisions, prepare a dispersion on the automatic muller of the reference sample of the coloured pigment as described in 6.1, 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. Clean the muller and spatula, and repeat the operation with the same quantities of the test sample of pigment and oil and using the same procedure on the same muller. Collect the coloured pigment paste from this sample and store. Clean the muller and spatula.

6.3 Mixing of dispersions of coloured pigment and white pigment

6.3.1 Place on the lower muller plate 3 ± 0,01 g of white pigment paste and an amount of the coloured pigment paste which has been ground for 200 revolutions which contains the mass of coloured pigment to give the chosen reduction ratio, and mix the two pastes with the spatula as homogeneously as possible, without grinding. Spread the mixture in an approximately circular patch in the centre of the lower plate, wipe the spatula on the upper plate, close the plates and grind for four stages of 25 revolutions each with the minimum applied force; after each stage collect the paste in the centre of the lower plate as previously described. Then remove the paste from the muller for later assessment and store in a suitable receptacle.

6.3.2 Repeat the operation using the coloured pigment dispersion paste prepared from the test sample of coloured pigment, thus producing a comparative reduction paste.

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

6.4.1 Place a quantity of each of the two reduction pastes obtained as described in 6.3 side by side on a glass plate or plastics film, and with the film applicator draw them down to form two uniformly thick strips not less than 25 mm wide with touching edges not less than 40 mm long. Compare for intensity of colour and for hue by examining the two strips in diffuse daylight through the glass or film of plastics material, and on the surface, immediately after application[1].

Where good daylight is not available, make the comparison in artificial daylight.

If the intensities of colour are equal and the hues are the same, the colours on reduction are the same, and the relative tinting strength of the sample under test is 100 % (see 7.4).

However, if the intensities of colour are equal but the hues are not the same, there is also a difference in colour on reduction; note the difference and its nature.

6.4.2 If the intensities of colour are not considered to be equal, and therefore the colours on reduction are not the same, repeat the operations of 6.3 and 6.4.1, but weighing and using a quantity of the coloured pigment dispersion of the test sample which is estimated to give an intensity of colour equal to that of the original mass taken of the coloured pigment dispersion of the reference sample.

For example, if it is considered that the sample under test is 15 % stronger than the reference sample, then in the repeat test, the mass of coloured test pigment dispersion used shall be 15 % less than that taken for the first test, but the mass of coloured pigment dispersion of the reference sample shall be the same as that originally used. If there is also a difference in hue, note the difference and its nature.

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1) If required the comparison may be carried out by using an instrumental method.

7 EXPRESSION OF RESULTS

7.1 Report the number of revolutions used.

7.2 Report the ratio of reduction used, i.e. 1 : 10, 1 : 15 or as appropriate.

7.3 Report the result of the comparison of the colour on reduction, if possible with a qualitative statement concerning any difference in hue.

7.4 Report the comparison of intensity of colour in linseed stand oil by one of the following methods, where a parts of the test sample produce the same intensity as b parts of the reference sample.

7.4.1 *Relative tinting strength* of the test sample is given by the formula :

$$\frac{b \times 100}{a} \text{ \% of the reference sample.}$$

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

7.4.2 *Equivalent colouring value* of the test sample is given by the formula :

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

NOTE — It is important to include the world "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.**

8 TEST REPORT

The test report shall include the following information :

a) a reference to ISO 787, Part XVI, or to a corresponding national standard.

b) type and identification of the pigment under test;

c) any deviation, by agreement or otherwise, from the test procedure described above;

d) the result of the test as indicated in 7.1, 7.2, 7.3 and 7.4;

e) the date of the test.