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# INTERNATIONAL STANDARD



# 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	Italy	Sweden
Egypt, Arab Rep. of	Netherlands	Switzerland
France	New Zealand	Turkey
Germany	Poland	United Kingdom
India	Romania	
Israel	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

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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 ( $\text{HgCl}_2$ ) 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

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3.1 Sintered silica crucible, porosity grade P10 or P16 (pore size index 4-16  $\mu\text{m}$ ).

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<sup>1)</sup> or the cold extraction method<sup>2)</sup>), 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

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<sub>4</sub>, as a percentage by mass, by the formula :

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

where

*m*<sub>0</sub> is the mass, in grams, of pigment used in the determination of matter soluble in water;

*m*<sub>1</sub> 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<sup>1)</sup> or the cold extraction method<sup>2)</sup> 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 :

$$0,1775 \frac{(V_1 - V_0)}{m}$$

where

*V*<sub>0</sub> is the volume, in millilitres, of 0,01 N silver nitrate solution required for the blank determination;

*V*<sub>1</sub> 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.

1) See Part III.  
2) See Part VIII.

**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<sup>1)</sup> or the cold extraction method<sup>2)</sup> 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<sub>3</sub>, as percentage by mass, by the formula :

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

where

*V*<sub>0</sub> is the volume, in millilitres, of ammonium chloride solution required by the blank determination;

*V*<sub>1</sub> 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.

# 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**<sup>1)</sup>.

3.6 **Conductivity cell**,<sup>1)</sup> 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.

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;

$\rho$  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  $\Omega$ ·m, see Figure).

NOTES

1 If a potassium chloride solution of different known concentration is used, the appropriate value of  $\rho$  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  $\rho_t$ , in ohm metres, of the aqueous extract of the pigment at the agreed temperature  $t$  °C, by the formula :

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

where

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

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

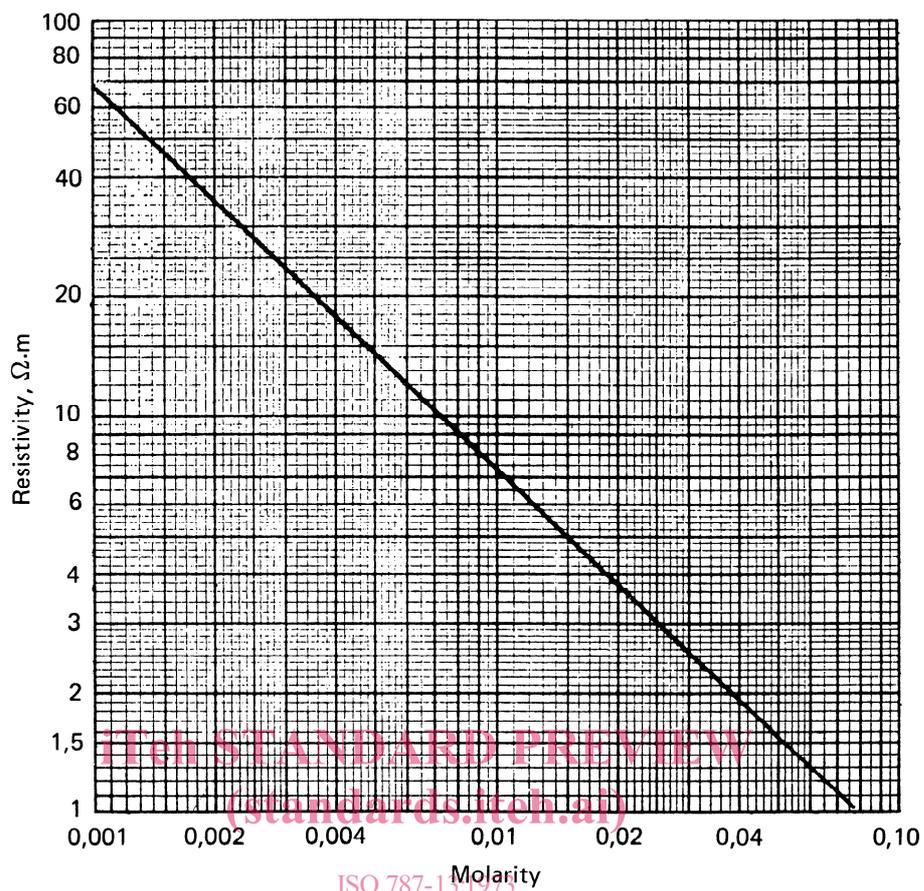
Express the result to the nearest 1 % of the value obtained.

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.

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 FIGURE 1 — 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 105/1, *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  $\mu\text{m}$ .

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

**3.4 Geometric grey scale**<sup>1)</sup> complying with Part 2 of ISO/R 105/1.

**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*.

1) Obtainable from most national standards institutions.