INTERNATIONAL STANDARD

ISO 787-14

Second edition
2002-08-01

General methods of test for pigments and extenders —

Part 14: Determination of resistivity of aqueous extract

Méthodes générales d'essai des pigments et matières de charge —

Partie 14: Détermination de la résistivité de l'extrait aqueux

Reference number
ISO 787-14:2002(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 787 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 787-14 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 2, *Pigments and extenders*.

This second edition cancels and replaces the first edition (ISO 787-14:1973), of which it constitutes a minor (editorial) revision.

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

- *Part 1: Comparison of colour of pigments*
- *Part 2: Determination of matter volatile at 105 °C*
- *Part 3: Determination of matter soluble in water — Hot extraction method*
- *Part 4: Determination of acidity or alkalinity of the aqueous extract*
- *Part 5: Determination of oil absorption value*
- *Part 7: Determination of residue on sieve — Water method — Manual procedure*
- *Part 8: Determination of matter soluble in water — Cold extraction method*
- *Part 9: Determination of pH value of an aqueous suspension*
- *Part 10: Determination of density — Pyknometer method*
- *Part 11: Determination of tamped volume and apparent density after tamping*
- *Part 13: Determination of water-soluble sulphates, chlorides and nitrates*
- *Part 14: Determination of resistivity of aqueous extract*
- *Part 15: Comparison of resistance to light of coloured pigments of similar types*
- *Part 16: Determination of relative tinting strength (or equivalent colouring value) and colour on reduction of coloured pigments — Visual comparison method*
- *Part 17: Comparison of lightening power of white pigments*
- *Part 18: Determination of residue on sieve — Mechanical flushing procedure*
- *Part 19: Determination of water-soluble nitrates (Salicylic acid method)*
- *Part 21: Comparison of heat stability of pigments using a stoving medium*
- *Part 22: Comparison of resistance to bleeding of pigments*

— *Part 23: Determination of density (using a centrifuge to remove entrained air)*

— *Part 24: Determination of relative tinting strength of coloured pigments and relative scattering power of white pigments — Photometric methods*

— *Part 25: Comparison of the colour, in full-shade systems, of white, black and coloured pigments — Colorimetric method*

General methods of test for pigments and extenders —

Part 14:
Determination of resistivity of aqueous extract

1 Scope

This part of ISO 787 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.

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, however.

The standard temperature of determination should preferably 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.

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 resistivity of aqueous extract be specified.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 787. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this part of ISO 787 are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Reagents

All reagents used shall be of recognized analytical reagent quality.

3.1 Conductivity water, resistivity not less than 2 500 $\Omega \cdot$ m.

3.2 Methanol, resistivity not less than 2 500 $\Omega \cdot$ m.

3.3 Potassium chloride, 0,02 mol/l solution.

4 Apparatus

4.1 Centrifuge, or ultra-centrifuge if necessary.

4.2 Filter paper, fine textured, washed with conductivity water on a filter funnel until the filtrate gives a resistivity greater than 2 000 $\Omega \cdot$ 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.

4.3 Cylinder, approximately 35 mm wide by 125 mm deep, or other container suitable for use with the conductivity cell to be employed.

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

4.5 Conductivity bridge.[1)]

4.6 Conductivity cell,[1)] having a cell constant K of approximately 1.

5 Sampling

The sample of pigment used for the test shall be taken in accordance with the provisions of ISO 15528.

6 Determination of cell constant

6.1 Prepare a working standard solution of potassium chloride by diluting the potassium chloride solution (3.3) with conductivity water to a known concentration.

Measure the resistance R of the working standard solution using the conductivity cell (4.6) at 23 °C (or at an alternative agreed temperature with appropriate corrections) as described in 7.2.2.

6.2 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 the KCl solution of the concentration used, in ohm metres (for a 0,002 mol/l solution, this is 34,4 $\Omega \cdot$ m — see Figure 1).

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

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 utilize the middle third of the scale.

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

7 Procedure

7.1 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 7.3. If the sample wets easily, proceed as described in 7.2.

7.2 Hydrophilic pigments

7.2.1 Add $(20 \pm 0{,}01)$ 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 test portion 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 a tendency to foam and crawl, can be handled better in a 400 ml beaker. A 20 g test portion 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.

7.2.2 Cool the filtrate to a temperature of about 20 °C. Rinse the cylinder (4.3) and the conductivity cell (4.6) 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 $(23 \pm 0{,}5)$ °C, using the conductivity bridge (4.5) 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.

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

7.3 Hydrophobic pigments

A modification of the procedure given in 7.2 is necessary for organic pigments that are not easily wetted with water.

Wet $(20 \pm 0{,}01)$ g of pigment with as much methanol (3.2) (4 g 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 g. Then proceed as outlined in 7.2.2.

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

8 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 equation:

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

where

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

K is the cell constant, determined in accordance with 6.2.