INTERNATIONAL STANDARD

ISO 787-13

Second edition
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General methods of test for pigments and extenders —

Part 13: Determination of water-soluble sulfates, chlorides and nitrates

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

Partie 13: Détermination des sulfates, chlorures et nitrates solubles dans l'eau

Reference number
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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

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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. 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 787-13 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 787-13:1973), 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 sulfates, chlorides and nitrates*

— *Part 14: Determination of resistivity of aqueous extract*

— *Part 15: Comparison of resistance to light of coloured pigments of similar types*

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

— *Part 17: Comparison of lightening power of white pigments*

— *Part 18: Determination of residue on sieve — 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 13:
Determination of water-soluble sulfates, chlorides and nitrates

1 Scope

This part of ISO 787 specifies a general method of test for determining the water-soluble sulfates, 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 the 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 sulfates chlorides or nitrates 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. Distilled water, or water of equivalent purity, shall be used.

3.1 Hydrochloric acid, $\rho = 1{,}18$.

3.2 Silver nitrate, 0,01 mol/l standard volumetric solution.

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

3.4 Sodium hydroxide solution, 200 g/l.

3.5 Barium chloride solution, 50 g/l.

3.6 Potassium chromate solution, 50 g/l.

3.7 Devarda's alloy, powdered.

3.8 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 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 over a period of several days, allow to stand, and use the clear supernatant liquid for the test.

4 Apparatus

Normal laboratory equipment, plus the following:

4.1 Sintered-silica filter crucible, porosity grade P10 or P16 (pores size index 4 μm to 16 μm).

4.2 Nessler cylinder, capacity 50 ml.

4.3 Distillation apparatus.

5 Sampling

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

6 Determination of sulfates

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)]), acidify with 3 ml of hydrochloric acid (3.1) and boil the solution vigorously, taking care to avoid loss of solution by splashing. Add barium chloride solution (3.5), drop by drop, to the hot solution until in slight excess, and allow the solution to stand overnight. Decant the supernatant liquid through the tared silica filter 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.

6.2 Expression of results

Calculate the water-soluble sulfate 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 sulfate precipitate.

Report the result to two decimal places.

1) See Part 3.

2) See Part 8.

7 Determination of chlorides

7.1 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 potassium chromate solution (3.6). Titrate with silver nitrate solution (3.2), 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 titrating with 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.

7.2 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 mol/l silver nitrate solution required for the blank determination;

V_1 is the volume, in millilitres, of 0,01 mol/l 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.

8 Determination of nitrates

8.1 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 (4.3) and dilute to 150 ml. Add 3 g of Devarda's alloy (3.7) and 30 ml of sodium hydroxide solution (3.4) and close the apparatus at once. Place 2 ml of hydrochloric acid (3.1) 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 (4.2). Dilute to 50 ml. Add 1 ml of Nessler's reagent (3.8) and match the colour against that of a similar standard solution prepared by adding ammonium chloride solution (3.3) from a burette.

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

8.2 Expression of results

Calculate the water-soluble nitrate content expressed as NO_3, as a 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.

9 Test report

The test report shall include the following information:

a) a reference to this part of ISO 787 (ISO 787-13);
b) all details necessary for complete identification of the pigment under test;
c) any deviation, by agreement or otherwise, from any of the test procedures 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 6.2, 7.2 or 8.2;
f) the date of the test.

ICS 87.060.10

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