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General methods of test for pigments and extenders —

Part 13: Determination of water-soluble sulphates, 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

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuffs and extenders*.

This third edition cancels and replaces the second edition (ISO 787-13:2002), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— Clause 3 on “Terms and definitions”, with a general reference to ISO 18451-1, has been added;

— CAS numbers have been added to the reagents;

— the bibliography has been added;

— the text has been editorially revised.

A list of all parts in the ISO 787 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

General methods of test for pigments and extenders —

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

1 Scope

This document specifies a general method of test for determining the water-soluble sulphates, chlorides and nitrates of pigments.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 18451-1, *Pigments, dyestuffs and extenders — Terminology — Part 1: General terms*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18451-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Reagents

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

4.1 Hydrochloric acid, ρ = 1,18 g/cm^3 (CAS-No 7647-01-0).

4.2 Silver nitrate, 0,01 mol/l standard volumetric solution (CAS-No 7761-88-8).

4.3 Ammonium chloride solution, 17,2 mg/l (CAS-No 12125-02-9).

4.4 Sodium hydroxide solution, 200 g/l (CAS-No 1310-73-2).

4.5 Barium chloride solution, 50 g/l (CAS-No 10361-37-2).

4.6 Potassium chromate solution, 50 g/l (CAS-No 7789-00-6).

4.7 Devarda's alloy, powdered.

4.8 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. 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.

5 Apparatus

Normal laboratory equipment, plus the following:

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

5.2 Nessler cylinder, capacity 50 ml.

5.3 Distillation apparatus.

6 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

7 Determination of sulphates

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]), acidify with 3 ml of hydrochloric acid (4.1) and boil the solution vigorously, taking care to avoid loss of solution by splashing. Add barium chloride solution (4.5), 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.

7.2 Expression of results

Calculate the water-soluble sulphate content $c(SO_4)$, as a percentage mass fraction, by Formula (1):

$$c(SO_4) = \frac{206\ m_1}{m_0} \quad (1)$$

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.

8 Determination of chlorides

8.1 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 (4.6). Titrate with silver nitrate solution (4.2), 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.

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

8.2 Expression of results

Calculate the water-soluble chloride content $c(\mathrm{Cl})$, as a percentage mass fraction, by Formula (2):

$$c(\mathrm{Cl}) = 0{,}177\ 5 \frac{(V_1 - V_0)}{m} \quad (2)$$

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.

9 Determination of nitrates

9.1 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 (5.3) and dilute to 150 ml. Add 3 g of Devarda's alloy (4.7) and 30 ml of sodium hydroxide solution (4.4) and close the apparatus at once. Place 2 ml of hydrochloric acid (4.1) 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 (5.2). Dilute to 50 ml. Add 1 ml of Nessler's reagent (4.8) and match the colour against that of a similar standard solution prepared by adding ammonium chloride solution (4.3) from a burette.

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

9.2 Expression of results

Calculate the water-soluble nitrate content $c(NO_3)$, as a percentage mass fraction, by Formula (3):

$$c(NO_3) = 0{,}5\frac{(V_1 - V_0)}{m} \tag{3}$$

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.

10 Test report

The test report shall include the following information:

a) all details necessary for complete identification of the pigment under test;

b) a reference to this document, i.e. ISO 787-13:2019;

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

d) the result of the test as indicated by 7.2, 8.2 or 9.2;

e) any deviation, by agreement or otherwise, from any of the test procedures described above;

f) the date of the test.

Bibliography

[1] ISO 787-3, *General methods of test for pigments and extenders — Part 3: Determination of matter soluble in water — Hot extraction method*

[2] ISO 787-8, *General methods of test for pigments and extenders — Part 8: Determination of matter soluble in water — Cold extraction method*