
INTERNATIONAL STANDARD



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Iron oxide pigments for paints

Pigments à base d'oxydes de fer pour peintures

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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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, International Standard ISO 1248 replaces ISO Recommendation R 1248-1970 drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*.

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The Member Bodies of the following countries approved the Recommendation :

Austria	India	Portugal
Brazil	Iran	South Africa, Rep. of
Chile	Israile	Spain
Denmark	Italy	Sweden
Egypt, Arab Rep. of	Netherlands	Switzerland
France	New Zealand	Turkey
Germany	Peru	United Kingdom
Greece	Poland	U.S.S.R.

No Member Body expressed disapproval of the Recommendation.

Iron oxide pigments for paints

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the requirements and corresponding test methods for all manufactured and natural iron oxide pigments, including micaceous iron oxide pigments and rapid dispersion pigments, in dry form, suitable for use in paints.

2 REFERENCES

ISO 787, *General methods of test for pigments*.

ISO 842, *Raw materials for paints and varnishes – Sampling*.

3 DESCRIPTION

Iron oxide pigments are pigments the colours of which – usually red, yellow, brown, black, or grey with a metallic sheen – are mainly due to iron oxides and hydrated iron oxides. The material may be in the form of a powder or in lamellar form (micaceous iron oxides).

4 CLASSIFICATION

4.1 General

In this International Standard, iron oxide pigments are classified

- by groups according to their colour;
- by categories according to their iron content expressed as iron(III) oxide;
- by types according to their content of water-soluble matter and their total content of water-soluble chlorides and sulphates expressed as the ions Cl^- and SO_4^{2-} ;
- by grades according to their residue on sieve;
- by classes according to their origin.

4.2 Definition of criteria for classification

4.2.1 Groups

According to their colour, iron oxide pigments are divided into five groups :

- reds
- yellows
- browns
- blacks
- greys with metallic sheen¹⁾.

4.2.2 Categories

According to their iron content, expressed as iron(III) oxide, iron oxide pigments are divided into the categories shown in table 1.

TABLE 1 – Categories

Group	Category	Minimum iron oxide content %
Red	A	95
	B	70
	C	50
	D	10
Yellow	A	83
	B	70
	C	50
	D	10
Brown	A	87
	B	70
	C	30
Black	A	95
	B	70
Grey with metallic sheen	A	85

1) This group comprises only micaceous pigments.

4.2.3 Types

According to their content of water-soluble matter and their total content of water-soluble chlorides and sulphates, iron oxide pigments are divided into the types shown in table 2.

TABLE 2 – Types

Characteristic	Type I ¹⁾	Type II		Type III
	Red and brown only	Red and brown	Yellow, black, and grey	
Matter soluble in water (determined after drying at 105 °C), % (m/m)	≤ 0,3	between 0,3 and 1	≤ 1	between 1 and 5
Sum of water-soluble chlorides and sulphates expressed as the ions Cl ⁻ and SO ₄ ²⁻ , % (m/m)	≤ 0,1			

1) Type 1 corresponds in particular to pigments used in making anti-corrosive paints.

4.2.4 Grades

According to their residue on sieve, iron oxide pigments are divided into the four grades shown in table 3.

TABLE 3 – Grades

Characteristic	Grade 1	Grade 2	Grade 3	Grade 4 grey only
Residue on sieve of mesh aperture 63 μm, % (m/m)	≤ 0,01	between 0,01 and 0,1	between 0,1 and 1	between 5 and 15

4.2.5 Classes

According to their origin, iron oxide pigments are divided into four classes :

- a) manufactured pigments without extenders;
- b) natural pigments without extenders;
- c) mixtures of natural and manufactured pigments without extenders;
- d) mixtures of pigments with extenders.

5 DESIGNATION

The designation of an iron oxide pigment shall include :

- a) an indication of the colour group to which it belongs, to which may be added a more precise indication of the actual colour (preferably by means of colorimetric data);
- b) its category;
- c) its type;
- d) its grade;
- e) its class;

NOTE – The following additional items may be included in the designation :

- the common name in some countries, especially for natural pigments (ochre, umber, (terra di) Sienna, micaceous pigment, etc.);
- an indication of its particular form (for example lamellar) or the treatment it has undergone (for example burnt, washed).

f) a reference to this International Standard or an equivalent national standard.

Examples:

Red iron oxide, A, I, 2, a, ISO 1248

Yellow iron oxide, D, II, 3, b, washed ochre, ISO 1248

Grey iron oxide with metallic sheen, A, I, 4, b, micaceous, ISO 1248

6 REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

Iron oxide pigments shall have the characteristics shown in table 4.

7 SAMPLING

7.1 A representative sample of the pigment shall be taken in accordance with ISO 842.

7.2 The sample agreed between the interested parties, to which reference is made at several points in table 4, shall be one and the same sample and shall comply with all the requirements specified for the pigment under test.

TABLE 4 — Required characteristics and their tolerances

Characteristic	Requirement according to group and category														Test method	
	Red				Yellow				Brown			Black		Grey		
	A	B	C	D	A	B	C	D	A	B	C	A	B	A		
Iron content, expressed as iron (III) oxide (Fe ₂ O ₃) (determined on the pigment after drying at 105 °C), % (m/m) min.	95	70	50	10	83	70	50	10	87	70	30	95	70	85	Clause 8	
Matter volatile at 105 °C, % (m/m) max.	≤ 0,3				2,5				2,5			1		2,5		ISO 787, Part II
Matter soluble in water, % (m/m)	Type I ≤ 0,3				Type II 0,3 to 1				Type III ≤ 1			1 to 5		≤ 1		ISO 787, Part III
Water-soluble chlorides and sulphates, expressed as ions Cl ⁻ and SO ₄ ²⁻ , % (m/m) max.	Type I 0,1				Type II 0,1				Type III 0,1			Type IV 0,1		Type V 0,1		ISO 787, Part XIII
Residue on sieve of mesh aperture 63 μm, % (m/m)	Grade 1 ≤ 0,01				Grade 2 0,01 to 0,1				Grade 3 0,1 to 1			Grade 4 5 to 15		Grade 5 20		ISO 787, Part VII
Acidity or alkalinity of aqueous extract, ml of 0,1 N solution, max.	Within ± 1,0 of that of the agreed sample														ISO 787, Part IV	
pH of aqueous suspension	Within + 15 % of that of the agreed sample														ISO 787, Part IX	
Oil absorption value	Negative test														ISO 787, Part V	
Lead chromate	Negative test														Clause 9	
Total calcium, expressed as calcium oxide, % (m/m) max.	Class a 0,3				Classes b and c 5				Class d To be agreed between the interested parties			To be agreed between the interested parties		Clause 10		
Colour	Equal to that of an agreed reference sample to within a tolerance fixed between the interested parties														ISO 787, Part I	
Relative tinting strength	Negative test														ISO 787, Part XVI	
Presence of organic colouring matter	Negative test														Clause 11	

METHODS OF TEST

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

8 DETERMINATION OF TOTAL IRON, EXPRESSED AS IRON(III) OXIDE

8.1 Reagents

8.1.1 Hydrochloric acid, ρ 1,18 g/ml.

8.1.2 Tin(II) chloride, 100 g/l solution.

Dissolve 50 g of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ in 300 ml of hydrochloric acid (8.1.1) and then dilute with water to 500 ml.

Keep the solution clear in a hermetically closed flask containing a little metallic tin.

8.1.3 Mercury(II) chloride, saturated solution (60 to 100 g/l).

8.1.4 Mixture of sulphuric and phosphoric acids

Mix 150 ml of sulphuric acid, ρ 1,84 g/ml, with 150 ml of phosphoric acid (H_3PO_4), ρ 1,70 g/ml at 85 %, and dilute with water to 1 l.

8.1.5 Diphenylamine, sulphuric acid solution.

Dissolve 1 g of barium diphenylaminesulphonate in 100 ml of sulphuric acid, ρ 1,84 g/ml.

8.1.6 Potassium dichromate, 0,1 N solution.

Dissolve 4,904 g of $\text{K}_2\text{Cr}_2\text{O}_7$, previously dried at 150 °C, in water and dilute to 1 l.

8.1.7 Ammonium thiocyanate, 170 g/l solution.

8.1.8 Potassium permanganate, 0,1 N solution.

8.2 Procedure

8.2.1 Test portion

Depending on the quantity of iron present in the sample, take 0,3 to 1,0 g of pigment and weigh to the nearest 0,1 mg.

8.2.2 Determination

Place the test portion in a 400 ml beaker and add 25 ml of hydrochloric acid (8.1.1).

NOTES

1 If the sample is known, or suspected, to contain organic matter, it shall be heated in a porcelain crucible until it reaches a dark red colour, before treating with acid.

2 Solution of the sample may be assisted by the addition of 0,5 g of potassium chlorate to the hydrochloric acid.

3 In the case of micaceous pigments, it is recommended to use 60 ml of the hydrochloric acid and 0,5 g of potassium chlorate.

Cover the beaker with a watch-glass and heat at 80 to 90 °C until all the dark particles in the insoluble residue disappear. To facilitate solution of the iron, add tin(II) chloride solution (8.1.2) a few drops at a time during the heating process without, however, removing completely the colour from the liquid. It is recommended that the liquid be stirred after each addition of tin(II) chloride solution. If too much tin chloride solution has been added in error, add some potassium permanganate to the solution until a yellow colour appears and then add tin(II) chloride solution, drop by drop, until the yellow colour disappears, subsequently adding one or two drops in excess. When the residue has become almost colourless, solution of the iron can be considered as complete.

NOTE – If a black insoluble residue remains, filter this residue and fuse it with sodium or potassium carbonate in a platinum crucible. Then extract with hydrochloric acid (8.1.1) and add the extract to the filtrate obtained previously.

Add 25 to 50 ml of water to the liquid and heat until gently boiling (avoid intense and prolonged boiling). While stirring, slowly add the tin(II) chloride solution drop by drop, until the last drop makes the solution colourless or free from yellow colour, then add one or two drops in excess. If too much tin(II) chloride solution has been added in error, add some potassium permanganate to the solution until a yellow colour appears and then add tin(II) chloride solution, drop by drop, until the yellow colour disappears, subsequently adding one or two drops in excess.

Test a drop of the solution on a spotting plate with a drop of ammonium thiocyanate solution (8.1.7) to confirm, by the absence of a red coloration, that the reduction of iron is complete.

Dilute with 200 ml of cold water, then add 15 ml of mercury(II) chloride solution (8.1.3) all at once, stirring vigorously. A slight white precipitate should appear after 15 to 20 s. One minute after having added the mercury(II) chloride, add 50 ml of the sulphuric/phosphoric acid mixture (8.1.4) and 3 drops of diphenylamine solution (8.1.5), and titrate slowly with the potassium dichromate solution (8.1.6) to an end-point when the dark green colour changes to violet. The titration shall be started not more than 3 min after adding the mercury(II) chloride solution.

8.3 Expression of results

Calculate the iron content of the sample, expressed as iron(III) oxide, as a percentage by mass, by the following formula :

$$0,7984 \frac{V}{m}$$

where

V is the volume, in millilitres, of the potassium dichromate solution used in the determination;

m is the mass, in grams, of the test portion.

Report the result to one decimal place.

9 TEST FOR PRESENCE OF LEAD CHROMATE

9.1 Reagents

9.1.1 Nitric acid, ρ 1,42 g/ml, diluted 1 + 5.

9.1.2 Potassium iodide, 100 g/l solution.

9.2 Procedure

Dissolve a portion of the test sample in cold nitric acid (9.1.1), stirring vigorously. Filter, then add to the filtrate a few millilitres of potassium iodide solution (9.1.2).

The appearance of yellow crystals indicates the presence of lead.

10 DETERMINATION OF TOTAL CALCIUM, EXPRESSED AS CaO

10.1 Reagents

10.1.1 Hydrochloric acid, ρ 1,18 g/ml, diluted 1 + 1.

10.1.2 Nitric acid, ρ 1,42 g/ml.

10.1.3 Methyl isobutyl ketone.

10.1.4 Ammonia solution, ρ 0,9 g/ml, free from carbon dioxide.

10.1.5 Methyl red, 1 g/l ethanolic solution.

10.1.6 Acetic acid, 99 to 100 %.

10.1.7 Ammonium oxalate, saturated solution.

10.1.8 Ammonium oxalate, 1 g/l solution.

10.1.9 Sulphuric acid, ρ 1,84 g/ml, diluted 1 + 4.

10.1.10 Potassium permanganate, 0,1 N solution, normality T .

10.2 Procedure

10.2.1 Test portion

Weigh in a flask $10 \pm 0,001$ g of the sample.

10.2.2 Determination

Dissolve the test portion in 100 ml of hydrochloric acid (10.1.1) to which 5 ml of nitric acid (10.1.2) have been added. Evaporate and dry on a sand bath.

Add 100 ml of hydrochloric acid (10.1.1), boil and after cooling decant into a 250 ml volumetric flask.

Make up to the graduation mark with the hydrochloric acid. Take 100 ml of this solution and evaporate to dryness.

Add 60 ml of the hydrochloric acid and extract the iron with 60 ml of methyl isobutyl ketone (10.1.3).

If the solution does not become colourless, extract a second time.

Recover the aqueous phase and neutralize with ammonia (10.1.4) in the presence of methyl red (10.1.5). Acidify the medium again with acetic acid (10.1.6), to be added slightly in excess.

Bring to the boil and add 50 ml of the ammonium oxalate solution (10.1.7) previously heated. Continue boiling until the precipitate becomes granular.

Leave to stand for approximately 1 h, filter and wash with the dilute ammonium oxalate solution (10.1.8) until the filtrate gives negative test for chloride.

Finish washing with the minimum of cold water to eliminate the ammonium oxalate.

Place the beaker in which the precipitation was carried out under the funnel, pierce the tip of the filter paper with a glass rod and wash the precipitate down into the beaker with hot water. Pour approximately 30 ml of warm sulphuric acid (10.1.9) on to the paper to wash it, and dilute the solution in the beaker to approximately 250 ml. Titrate at about 75 °C with the potassium permanganate solution (10.1.10) (the temperature of the solution should not be lower than 60 °C at the end of the titration).

10.3 Expression of results

Calculate the calcium content, expressed as calcium oxide (CaO), as a percentage by mass, by the following formula :

$$0,701 VT$$

where

V is the volume, in millilitres, of potassium permanganate solution used;

T is the normality of the potassium permanganate solution.

Report the result to one decimal place.

11 TEST FOR ORGANIC COLOURING MATTER

11.1 Reagents

11.1.1 Ethanol, 95 % (V/V).

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11.1.2 Sodium hydroxide, 1 N ethanolic solution.

11.1.3 Chloroform.

11.2 Procedure

11.2.1 Test portion

Place about 2 g of sample in each of two test tubes.

11.2.2 Determination

Boil one of the test portions with 25 ml of water, allow to settle and decant the supernatant liquid.

Boil the residue with 25 ml of ethanol (11.1.1) and decant as before.

Boil the residue with 25 ml of sodium hydroxide solution (11.1.2) and again decant.

Boil the other test portion with 25 ml of chloroform (11.1.3), allow to settle and decant the supernatant liquid.

Note the colours of the supernatant liquids.

11.3 Expression of results

Report the presence of organic colouring matter, if one of the above solutions is coloured.

If the solutions remain uncoloured, there is probably no organic colouring matter present.

NOTE — The presence of organic colouring matter may also be detected by a characteristic smell on calcination.

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