INTERNATIONAL STANDARD

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Iron oxide pigments — Specifications and methods of test

Pigments à base d'oxydes de fer — Spécifications et méthodes d'essai

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1248 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 1248:1974), which has been technically revised.

Iron oxide pigments — Specifications and methods of test

1 Scope

This International Standard specifies the requirements and the corresponding methods of test for all manufactured and natural iron oxide pigments, in dry form, suitable for general use. These pigments are identified by Colour Index Nos.[1)] red 101 and 102, yellow 42 and 43, brown 6 and 7 and black 11, and includes “rapid-dispersion pigments”.

This International Standard does not cover micaceous iron oxide pigments (see Note 1), transparent iron oxide pigments, granular grey iron oxide (see Note 2) or magnetic iron oxide pigments other than those of Colour Index Pigment black 11.

NOTE 1 The requirements and the corresponding methods of test for micaceous iron oxide pigments are specified in ISO 10601.

NOTE 2 Granular grey iron oxides are too abrasive for general use.

2 Normative references

The following referenced documents are indispensable for the application 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 385–1, *Laboratory glassware — Burettes — Part 1: General requirements*

ISO 648, *Laboratory glassware — One-mark pipettes*

ISO 787-1, *General methods of test for pigments and extenders — Part 1: Comparison of colour of pigments*

ISO 787-2, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C*

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

ISO 787-4, *General methods of test for pigments and extenders — Part 4: Determination of acidity or alkalinity of the aqueous extract*

ISO 787-5, *General methods of test for pigments and extenders — Part 5: Determination of oil absorption value*

ISO 787-7, *General methods of test for pigments and extenders — Part 7: Determination of residue on sieve — Water method — Manual procedure*

1) The Colour Index is published by The Society of Dyers and Colourists, PO Box 244, Perkin House, 82 Grattan Road, Bradford, West Yorkshire BD1 2JB, United Kingdom; and the American Association of Textile Chemists and Colorists, National Headquarters, Box 12215, Research Triangle Park, N.C. 27709, USA.

ISO 787-9, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension*

ISO 787-13, *General methods of test for pigments and extenders — Part 13: Determination of water-soluble sulfates, chlorides and nitrates*

ISO 787-16, *General methods of test for pigments and extenders — Part 16: Determination of relative tinting strength (or equivalent colouring value) and colour on reduction of coloured pigments — Visual comparison method*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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

3 Description

The iron oxide pigments covered by this International Standard consist mainly of iron oxides and hydrated iron oxides. Their colours are usually red, yellow, brown or black.

4 Classification

4.1 General

In this International Standard, iron oxide pigments are classified as follows:

— by **groups** depending on their colour;

— by **categories** depending on their iron content, expressed as iron(III) oxide;

— by **types** depending on their content of water-soluble matter and their total content of water-soluble chlorides and sulfates, expressed as the ions Cl^- and SO_4^{2-};

— by **grades** depending on their residue on sieve;

— by **classes** depending on their origin.

4.2 Criteria for classification

4.2.1 Groups

Depending on their colour, iron oxide pigments are divided into four groups:

— reds;

— yellows;

— browns;

— blacks.

4.2.2 Categories

Depending on their minimum iron content, expressed as iron(III) oxide, iron oxide pigments are divided into the categories shown in Table 1.

Table 1 — Categories of iron oxide pigments

Group	Category	Minimum iron content, expressed as Fe_2O_3 % (by mass)	Colour Index No.
Red	A	95	Pigment red 101 77491
	B C D	70 50 10	Pigment red 102 77491
Yellow	A	83	Pigment yellow 42 77492
	B C D	70 50 10	Pigment yellow 43 77492
Brown	A	87	Pigment brown 6 77491, 77492 or 77499
	B C	70 30	Pigment brown 7 77491, 77492 and/or 77499
Black	A B	95 70	Pigment black 11 77499

4.2.3 Types

Depending on their content of water-soluble matter and their total content of water-soluble chlorides and sulfates, iron oxide pigments are divided into the types shown in Table 2.

Table 2 — Types of iron oxide pigments

Characteristic	Type I[a]	Type II		Type III	Method of test
	Red and brown only	Red and brown only	Yellow and black only	All pigments	
Matter soluble in water (determined after drying at 105 °C), % (by mass)	$\leqslant$ 0,3	> 0,3 and $\leqslant$ 1	$\leqslant$ 1	> 1 and $\leqslant$ 5	ISO 787-3
Sum of water-soluble chlorides and sulfates, expressed as the ions Cl^- and SO_4^{2-}, % (by mass)	$\leqslant$ 0,1				ISO 787-13

[a] Type I pigments are used in making anticorrosive paints.

4.2.4 Grades

Depending on their residue on sieve, iron oxide pigments are divided into the grades shown in Table 3.

Table 3 — Grades of iron oxide pigments

Characteristic	Grade 1	Grade 2	Grade 3	Method of test
	Red, yellow, brown and black			
Residue on sieve (45 µm), % (by mass)	⩽ 0,01	> 0,01 and ⩽ 0,1	> 0,1 and ⩽ 1	ISO 787-7

4.2.5 Classes

Depending on their origin, iron oxide pigments are divided into four classes:

— class a manufactured pigments without extenders;

— class b natural pigments without extenders;

— class c mixtures of natural and manufactured pigments without extenders;

— class d mixtures of pigments with extenders.

For classes a, b and c, the permitted maximum content of calcium (as CaO) is shown in Table 4.

5 Designation

The designation of an iron oxide pigment shall include the following.

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).

The following additional items may be included in this part of the designation:

— the common name in some countries, especially for natural pigments [ochre, umber, (terra di) Sienna, etc.];

— an indication of the treatment it has undergone (e.g. burnt, washed).

b) A reference to this International Standard, i.e. ISO 1248.

c) The category of the iron oxide pigment.

d) Its type.

e) Its grade.

f) Its class.

EXAMPLES

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

Yellow iron oxide (washed ochre) ISO 1248-D-II-3-b.

6 Required characteristics and associated tolerances

6.1 For iron oxide pigments complying with this International Standard, the essential requirements are specified in Table 4 and the conditional requirements are listed in Table 5. The conditional requirements shall be specified by agreement between the interested parties.

6.2 The agreed reference pigment referred to in Table 5 shall comply with the requirements of Table 4.

7 Sampling

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

Table 4 — Essential requirements

Characteristic		Requirement depending on group and category													Method of test
		Red				Yellow				Brown			Black		
		A	B	C	D	A	B	C	D	A	B	C	A	B	
Total iron, expressed as iron(III) oxide (Fe_2O_3), determined on the pigment after drying at 105 °C, % (by mass) min.		95	70	50	10	83	70	50	10	87	70	30	95	70	See Clause 8
Matter volatile at 105 °C, % (by mass) max.		1	1,5	2,5	2,5	1	2,5	2,5	2,5	1	2,5	2,5	1	2,5	ISO 787-2
Matter soluble in water (hot extraction method), % (by mass)	Type I	⩽ 0,3				—				⩽ 0,3			—		ISO 787-3
	Type II	> 0,3 and ⩽ 1				⩽ 1				> 0,3 and ⩽ 1			⩽ 1		
	Type III	> 1 and ⩽ 5													
Water-soluble chlorides and sulfates, expressed as ions Cl^- and SO_4^{2-}, % (by mass)	Type 1	⩽ 0,1				—				⩽ 0,1			—		ISO 787-13
Residue on sieve (45 µm), % (by mass)	Grade 1	⩽ 0,01													ISO 787-7
	Grade 2	> 0,01 and ⩽ 0,1													
	Grade 3	> 0,1 and ⩽ 1													
Acidity or alkalinity of aqueous extract solution, ml of 0,1 mol/l max.		20													ISO 787-4
Presence of lead chromate		Not detectable													See Clause 9
Total calcium, expressed as calcium oxide, determined on the pigment after drying at 105 °C, % (by mass) max.	Class a	0,3													See Clause 10
	Classes b and c	5													
	Class d	See Table 5													
Presence of organic colouring matter		Not detectable													See Clause 11

Table 5 — Conditional requirements for all groups and categories

Characteristic		Requirement	Method of test
pH value of the aqueous suspension		Shall not differ by more than 1 pH scale unit from that of the agreed reference pigment (see 6.2)	ISO 787-9
Oil absorption value		Shall not differ by more than 15 % from that of the agreed reference pigment (see 6.2)	ISO 787-5
Total calcium expressed as calcium oxide, % (by mass) max.	Class a	See Table 4	See Clause 10
	Classes b and c		
	Class d	To be agreed between the interested parties	
Colour		Equal to that of the agreed reference pigment (see 6.2) within a tolerance agreed between the interested parties	ISO 787-1
Relative tinting strength			ISO 787-16

8 Determination of total iron, expressed as iron(III) oxide (Fe_2O_3)

For the determination of the total iron content, two methods are provided. Method A (8.1) shall be used as the reference method in cases of dispute.

It is recommended that mercury be removed from the waste solutions before discharge to effluent drains. A suggested procedure is given in Annex A.

8.1 Method A

8.1.1 Principle

A test portion of the dried sample is dissolved in hydrochloric acid. The iron(III) is then reduced to iron(II) with tin(II) chloride solution, and the excess reducing agent is oxidized with mercury(II) chloride solution, followed by titration of the iron(II) with potassium dichromate solution, using sodium diphenylamine sulfonate as indicator.

8.1.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity as defined in ISO 3696.

WARNING — Use the reagents in accordance with the appropriate health and safety regulations.

8.1.2.1 **Hydrochloric acid**, concentrated, approximately 37 % (by mass), $\rho \approx 1{,}19$ g/ml.

8.1.2.2 **Hydrochloric acid**, diluted 1 + 50.

Add 1 part by volume of concentrated hydrochloric acid (8.1.2.1) to 50 parts by volume of water.

8.1.2.3 **Hydrofluoric acid**, concentrated, approximately 40 % (by mass), $\rho \approx 1{,}13$ g/ml.

8.1.2.4 **Sulfuric acid**, diluted 1 + 1.

Add carefully 1 part by volume of concentrated sulfuric acid [approximately 96 % (by mass), $\rho \approx 1{,}84$ g/ml] to 1 part by volume of water.