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Micaceous iron oxide pigments for paints — Specifications and test methods

Pigments d'oxyde de fer micacé pour peintures — Spécifications et méthodes d'essai

Reference number
ISO 10601:1993(E)

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.

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.

International Standard ISO 10601 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Sub-Committee SC 2, *Pigments and extenders*.

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Introduction

Micaceous iron oxide pigments were previously included in ISO 1248:1974 and classified in the colour group "grey with metallic sheen". In this new International Standard, the requirements for micaceous iron oxide pigment have been defined more clearly and include assessment of particle shape.

Micaceous iron oxide pigments can vary in composition, particle size range, and particle shape depending on whether they are produced synthetically or, if a refined natural oxide, on the location where the ore was mined.

The primary use of micaceous iron oxide is in protective coatings for steelwork, and for optimum performance the pigment should have a high content of thin, flake-like particles. The protective action is ascribed to the close packing of pigment platelets within the paint film, forming overlapping layers that lie roughly parallel to the substrate. This impedes penetration of corrosion promoters, reduces ultra-violet degradation of the binder and improves film strength. For less critical requirements, micaceous iron oxide pigment with a lower content of thin flakes may be acceptable. Therefore in this International Standard, micaceous iron oxide pigments are classified into three groups according to their thin-flake content as determined by microscopic examination. (See table 1.)

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Micaceous iron oxide pigments for paints — Specifications and test methods

1 Scope

This International Standard specifies the requirements and corresponding test methods for manufactured and natural micaceous iron oxide (MIO) pigments, in dry form, used primarily in protective coatings for steelwork.

In accordance with current practice, the general requirements for micaceous iron oxide pigments have been sub-divided to give

a) those requirements that are essential (see table 2)

and

b) those requirements that are conditional upon prior agreement between the interested parties (see table 3).

In certain instances, reference may be made to an agreed reference pigment.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 150:1980, *Raw, refined and boiled linseed oil for paints and varnishes — Specifications and methods of test.*

ISO 787-2:1981, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C.*

ISO 787-3:1979, *General methods of test for pigments and extenders — Part 3: Determination of matter soluble in water — Hot extraction method.*

ISO 787-5:1980, *General methods of test for pigments and extenders — Part 5: Determination of oil absorption value.*

ISO 787-7:1981, *General methods of test for pigments and extenders — Part 7: Determination of residue on sieve — Water method — Manual procedure.*

ISO 787-9:1981, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension.*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

ISO 1248:—[1], *Iron oxide pigments (except micaceous) — Specifications and methods of test.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 micaceous iron oxide pigment: A refined mineral (also known as specular haematite) or a manufactured product consisting essentially of iron(III) oxide, Fe_2O_3. It is grey in colour with a metallic sheen and has a more or less lamellar form.

4 Classification

In this International Standard, micaceous iron oxide pigments are classified by grades according to their thin-flake content as shown in table 1 and by types according to their residue on sieve as shown in table 2.

1) To be published. (Revision of ISO 1248:1974)

Thin-flake micaceous iron oxide particles are defined as having a thickness such that they appear as sharply defined red translucent platelets when viewed under an optical microscope by transmitted light, i.e. with the light source behind the specimen under examination (see test method in clause 7).

Table 1 — Lamellar classification

Grade	Thin-flake content %
1	> 50
2	10 to 50
3	< 10

5 Required characteristics and their tolerances

5.1 For micaceous iron oxide pigments complying with this International Standard, the essential requirements are specified in tables 1 and 2 and the conditional requirements are referred to in table 3.

5.2 The reference pigment and the conditional requirements set out in table 3 shall be the subject of agreement between the interested parties.

5.3 The reference pigment shall comply with the requirements given in table 1 (grade 1, 2 or 3) and table 2 (type 1, 2 or 3).

6 Sampling

Take a representative sample of the product to be tested, as described in ISO 842.

7 Assessment of thin-flake content

7.1 Reagent

7.1.1 Refined linseed oil, complying with ISO 150.

Table 2 — Essential requirements

Characteristic		Requirement for MIO of type 1	2	3	Test method
Iron content, expressed as iron(III) oxide (Fe_2O_3) (determined on the pigment after drying at 105 °C)	% (*m/m*)	min. 85			ISO 1248[1]
Matter volatile at 105 °C	% (*m/m*)	max. 0,5			ISO 787-2
Matter soluble in water (hot-extraction method)	% (*m/m*)	max. 0,5			ISO 787-3
Residue on sieve	% (*m/m*)				ISO 787-7
	63 µm	max. 5	max. 15	max. 35	
	105 µm	max. 0,1[2]	max. 0,1	max. 0,1	

1) The use of 60 ml of 37 % (*m/m*) hydrochloric acid, ϱ approximately 1,19 g/ml, and 0,5 g of potassium chlorate is recommended to facilitate dissolution of the sample.

2) Or less by agreement between the interested parties.

Table 3 — Conditional requirements

Characteristic	Requirement	Test method
pH of aqueous suspension	Shall not differ by more than 1 pH unit from that of the agreed reference pigment (see 5.2)	ISO 787-9
Oil absorption value	Shall not differ by more than ± 15 % from that of the agreed reference pigment (see 5.2)	ISO 787-5
Total calcium, expressed as calcium oxide % (*m/m*)	To be agreed between the interested parties	ISO 1248

7.2 Apparatus

7.2.1 Glass microscope slide, approximately 25 mm × 75 mm × 1 mm, and cover slip.

7.2.2 Glass rod, approximately 100 mm long and 5 mm in diameter, with rounded end.

7.2.3 Optical microscope, capable of ×200 magnification, with a ×20 objective lens and fitted with high-intensity sub-stage illumination.

Use of a "wide-field" eye-piece is recommended, preferably incorporating a frame-mask or graticule so that a rectangular frame image appears in the field of view.

7.3 Procedure

Prepare a microscope slide (7.2.1) by one of the following methods:

a) Place a few milligrams of a representative sample of the dry pigment (see clause 6) on the glass slide. Add a few drops of refined linseed oil (7.1.1) and gently incorporate the pigment into the oil using the glass rod (7.2.2). Place the cover slip over the pigment/oil dispersion and position on the microscope stage.

b) Spread a few milligrams of the sample of the dry pigment on the glass slide and examine it in the way described below without adding linseed oil.

Adjust the intensity of the light below the slide to the highest practical level. Focus sharply at ×200 magnification and scan the slide until a field of view is obtained that is considered to be representative, and in which at least 50 particles can be seen.

Thin-flake micaceous iron oxide particles appear as sharply defined red translucent platelets whilst thicker and/or granular particles appear as black shapes.

Assess the proportion of red to black particles and classify as the appropriate grade (see table 4).

Table 4 — Thin-flake content

Grade	Appearance	Thin-flake content %
1	Greater proportion of red particles	> 50
2	Greater proportion of black particles but not all black	10 to 50
3	All black particles or only a few red particles	< 10

If the thin-flake content is not obvious, count the number of red and black particles in a group of at least 50 particles. The recommended frame-image device (see 7.2.3) assists this operation. Repeat the count on a duplicate slide and calculate the mean percentage of red particles. To assist counting, it may be helpful to use an eye-piece pointer [see for example ASTM D 1030-76 (Reapproved 1990), *Standard Test Method for Fiber Analysis of Paper and Paperboard*].

Typical photographs of the three grades of micaceous iron oxide pigment viewed with an optical microscope by transmitted light are shown in figures 1 a), 1 b) and 1 c). For comparison purposes, scanning electron photomicrographs of the three grades are shown in figures 1 d), 1 e) and 1 f).

8 Test report

The test report shall contain at least the following information:

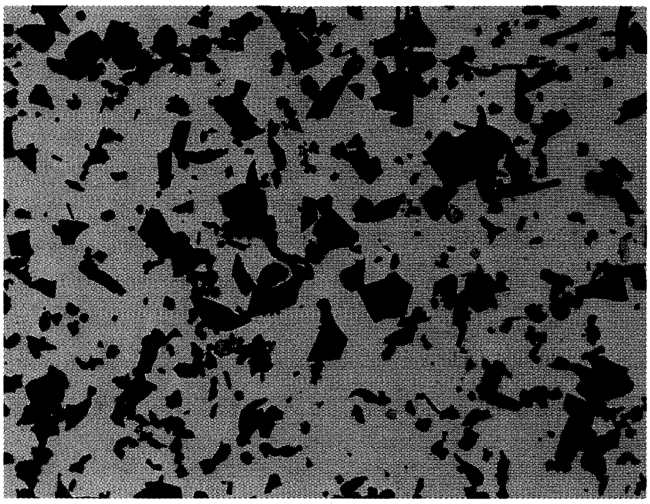
a) all details necessary for the identification of the product tested;

b) a reference to this International Standard (ISO 10601);

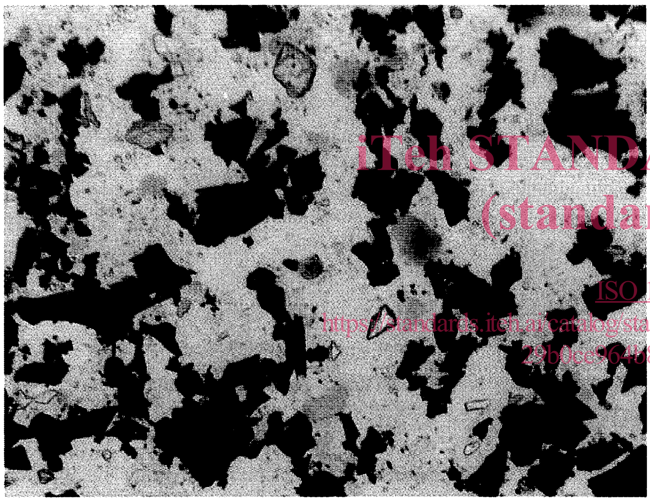
c) the results of the tests and whether or not the product complies with the relevant specification limits;

d) any deviation from the test methods specified;

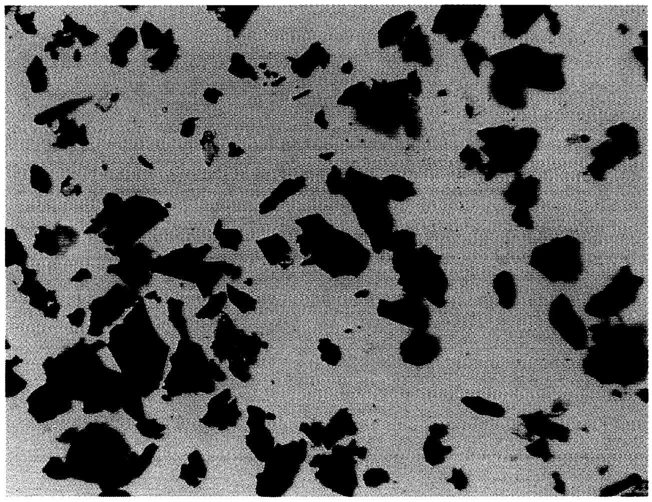
e) the date of the tests.



a) Grade 1, optical microscope (transmitted light)

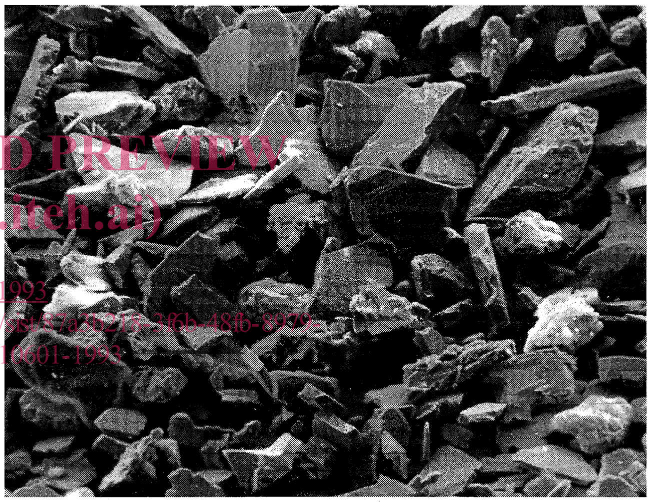


b) Grade 2, optical microscope (transmitted light)

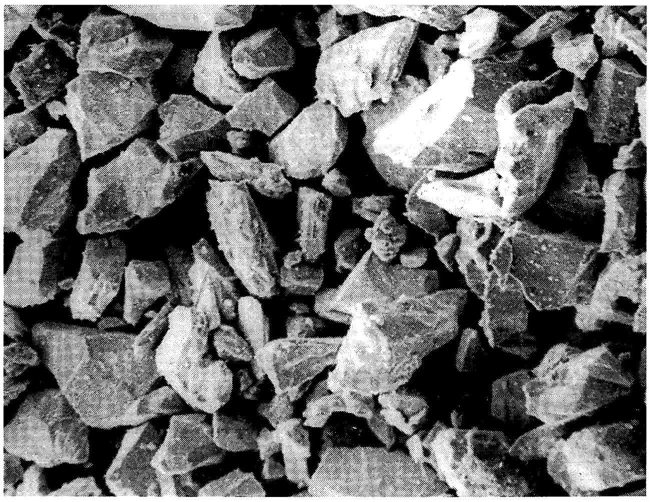


c) Grade 3, optical microscope (transmitted light)

d) Grade 1, scanning electron microscope



e) Grade 2, scanning electron microscope



f) Grade 3, scanning electron microscope

Figure 1 — Typical appearance of the three grades of micaceous iron oxide pigment at ×200 magnification

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