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ISO RECOMMENDATION

R 1248

IRON OXIDE PIGMENTS

1st EDITION

July 1970

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BRIEF HISTORY

The ISO Recommendation R 1248, *Iron oxide pigments,* was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes,* the Secretariat of which is held by the Nederlands Normalisatie-Instituut (NNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1248, which was circulated to all the ISO Member Bodies for enquiry in October 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

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ISO Recommendation R 1248 July 1970

IRON OXIDE PIGMENTS

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1. SCOPE

This ISO Recommendation specifies the requirements and the corresponding methods of test applying to all manufactured and natural iron oxide pigments, including micaceous iron oxide pigments and rapid dispersion pigments, in dry form.

2. DESCRIPTION

The pigments should be in the form of powder except for micaceous iron oxides which should be in lamellar form. Their colours – usually reds, yellows, browns, blacks, or greys with a metallic sheen – are mainly due to iron oxides and hydrated iron oxides.

The pigments should be free from organic colouring matter.

3. CLASSIFICATION

3.1 General

In this ISO Recommendation, 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 a sieve;
- by classes according to their origin.

3.2 Definition of criteria for classification

3.2.1 *Groups*. According to their colour, iron oxide pigments are divided into five groups :

- reds
- yellows
- browns
- blacks
- greys with metallic sheen*

* This group comprises only micaceous pigments.

3.2.2 *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

3.2.3 *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

	Type I*	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)	⩽ 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-}	⩽ 0.1 %			

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

3.2.4 *Grades.* According to their residue on a sieve, iron oxide pigments are divided into the four grades shown in Table 3.

TABLE 3 – Grades

	Grade 1	Grade 2	Grade 3	Grade 4 grey only
Residue on sieve of mesh aperture 63 μm	⩽ 0.01 %	between 0.01 % and 0.1 %	between 0.1 % and 1 %	between 5 % and 15 %

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3.2.5 *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.

4. DESIGNATION

The designation of an iron oxide pigment should include :

(1) 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);

(2) its category;

(3) its type;

(4) its grade;

(5) 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).

(6) a reference to this ISO Recommendation or an equivalent national standard.

Examples :

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

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

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

5. REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

Iron oxide pigments should have the characteristics shown in Table 4.

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TABLE 4 – Required characteristics

Characteristic		Requirements 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 (determined on the pigment after drying at 105 °C), % minimum		95	70	50	10	83	70	50	10	87	70	30	95	70	85	clause 7.1
Matter volatile at 105 °C, % maximum		1	1.5	2.5	2.5	1	2.5	2.5	2.5	1	2.5	2.5	1	2.5	1	ISO/R 787, Part II
Matter soluble in water, %	Type I	⩽ 0.3								⩽ 0.3						ISO/R 787, Part III
	Type II	0.3 to 1				⩽ 1				0.3 to 1			⩽ 1		⩽ 1	
	Type III	1 to 5														
Water-soluble chlorides and sulphates expressed as ions Cl^- and SO_4^{2-}, % maximum	Type I	0.1								0.1						ISO/R 787, Part . . . *
Residue on sieve, % (aperture 63 μm)	Grade 1	⩽ 0.01														ISO/R 787, Part VII
	Grade 2	0.01 to 0.1														
	Grade 3	0.1 to 1														
	Grade 4														5 to 15	
Acidity or alkalinity of aqueous extract, maximum		20 ml of 0.1 N solution														ISO/R 787, Part IV
pH of aqueous suspension		within ± 1.0 of that of the agreed sample														ISO/R 787, Part IX
Oil absorption value		within ± 15 % of that of the agreed sample														ISO/R 787, Part V
Lead chromate		negative test														clause 7.2
Total calcium expressed as calcium oxide, % maximum	Class a	0.3													to be agreed between parties	clause 7.3
	Classes b and c	5														
	Class d	to be agreed between parties														
Colour		equal to that of an agreed reference sample to within a tolerance fixed between parties														ISO/R 787, Part I
Relative tinting strength																ISO/R 787, Part . . . *
Presence of organic colouring matter		negative test														clause 7.4

* Under study

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6. SAMPLING

6.1 A representative sample of the pigment should be taken in accordance with ISO Recommendation R 842, *Sampling raw materials for paints and varnishes.*

6.2 The sample agreed between purchaser and vendor, to which reference is made at several points in Table 4, should be one and the same sample and should comply with all the requirements specified for the pigment under test.

7. METHODS OF TEST

NOTE. – All reagents used should be of recognized analytical reagent quality. Distilled water or water of equivalent purity should be used.

7.1 Determination of total iron (expressed as iron(III) oxide)

7.1.1 *Reagents*

(1) *Hydrochloric acid* ($d = 1.18$).

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

Dissolve 50 g of $SnCl_2.2H_2O$ in 300 ml of hydrochloric acid (1) and then dilute with water to 500 ml.

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

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

(4) *A mixture of sulphuric and phosphoric acid.*

Mix 150 ml of sulphuric acid ($d = 1.84$) with 150 ml of phosphoric acid H_3PO_4 ($d = 1.70$ at 85 %) and dilute with water to 1 litre.

(5) *Diphenylamine* , sulphuric acid solution.

Dissolve 1 g of barium diphenylaminesulphonate in 100 ml of sulphuric acid ($d = 1.84$).

(6) *Potassium dichromate*, 0.1 N solution.

Dissolve 4.904 g of $K_2Cr_2O_7$, previously dried at 150 °C, in water and dilute to 1 litre.

(7) *Ammonium thiocyanate*, 170 g/l solution.

(8) *Potassium permanganate*, 0.1 N solution.

7.1.2 *Procedure*

7.1.2.1 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.

7.1.2.2 DETERMINATION. Place the test portion in a 400 ml beaker and add 25 ml of hydrochloric acid (1).

NOTES

1. If the sample is known, or suspected, to contain organic matter, it should 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 watchglass 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 chloride solution (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 chloride solution*. 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 (1) and add the extract to the filtrate obtained previously.

* If too much tin chloride has been added in error, add some potassium permanganate to the solution until a yellow colour appears and then add tin chloride solution, drop by drop, until the yellow colour disappears, subsequently adding one or two drops in excess.

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