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Iron blue pigments for paints

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FOREWORD

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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.

International Standard ISO 2495 was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes.*

It was approved in September 1971 by the Member Bodies of the following countries :

Austria	Italy	Sweden
Egypt, Arab Rep. of	Netherlands	Switzerland
Germany	New Zealand	Turkey
India	Poland	United Kingdom
Israel	Romania	U.S.S.R.

No Member Body expressed disapproval of the document.

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Iron blue pigments for paints

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the requirements and the corresponding methods of test for iron blue pigments for paints.

2 REFERENCES

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

ISO/R 842, *Sampling raw materials for paints and varnishes.*

3 DESCRIPTION

Iron blue pigment is a pigment formed by the reaction of iron salts with cyanoferrate(II) or (III), if necessary followed by a treatment with oxidizing agents (for identification, see 6.1).

The material shall be in the form of a soft dry powder or in such a condition that it may be readily reduced thereto by crushing under a palette knife, without grinding action.

NOTE — The pigment shall be free from added colouring matter and from admixture of any substances other than those added during manufacture for the purpose of improving the quality or the working properties, or both, of the pigment.

4 REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

Iron blue pigments for paints shall have the characteristics shown in the following Table.

TABLE — **Required characteristics and their tolerances**

Characteristic	Requirement	Test method
Colour	Shall closely match that of the agreed sample	ISO/R 787 Part I
Colour on reduction		ISO/R 787 Part XVI
Relative tinting strength		ISO/R 787 Part XVI
Matter volatile at 60 °C	Can vary with the type of iron blue, but in no case shall exceed 4.0 %	clause 6.2[1)]
Matter soluble in water; hot extraction method	Can vary with the type of iron blue, but in no case shall exceed 2.0 %	ISO/R 787 Part III
Acidity or alkalinity of aqueous extract	Maximum 20 ml of 0.1 N solution per 100 g of pigment	clause 6.3
Oil absorption value	Shall not differ by more than 10 % from the value agreed between purchaser and vendor	ISO/R 787 Part V
Ease of dispersion	Shall not be inferior to that of the agreed sample	clause 6.4

1) The method given in ISO/R 787 Part II (heating at 105 °C) is not suitable for iron blue pigments, because water of crystallization tends to be lost at the higher temperature and reproducible results are therefore not obtained.

5 SAMPLING

5.1 A representative sample of the pigment shall be taken in accordance with ISO/R 842.

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

6 METHODS OF TEST

6.1 Identification

6.1.1 *Reagents*

1) **Sodium hydroxide** solution, 50 g/l.

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

3) **Iron(III) sulphate** solution, 20 g/l.

6.1.2 *Procedure*

To approximately 0.1 g of pigment in a 50 ml beaker, add 15 ml of the sodium hydroxide solution. Heat to boiling. In a few minutes the blue colour should be completely destroyed, and the characteristic reddish brown precipitate or iron(III) hydroxide should appear. Add the hydrochloric acid until faintly acid to litmus. The iron blue should be reformed, yielding again the characteristic blue colour.

NOTE — If the sodium hydroxide treatment does not completely destroy the blue colour, there is strong evidence that a foreign pigment is present. If this occurs, it is best to filter the alkaline solution, weakly acidify the filtrate with hydrochloric acid and add 2 ml of the iron(III) sulphate solution. The formation of a blue precipitate establishes the pigment as consisting, at least in part, of iron blue.

6.2 Matter volatile at 60 °C

6.2.1 *Procedure*

Weigh, to the nearest 1 mg, such a quantity of the pigment into a weighing bottle of about 65 mm diameter that the depth of the uniform layer of the pigment does not exceed 5 mm.

Heat the weighing bottle and contents for 16 h (overnight, for example) at 60 ± 2 °C. Cool in a desiccator containing dry silica gel and reweigh.

6.2.2 *Expression of results*

Calculate the matter volatile at 60 °C, as a percentage by mass, by the formula :

$$\frac{m_1 - m_2}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the weighing bottle and test portion before heating;

m_2 is the mass, in grams, of the weighing bottle and test portion after heating.

Report the result to the nearest 0.1 % (*m/m*).

6.3 Acidity or alkalinity of the aqueous extract

6.3.1 *Procedure*

Take 100 ml of the filtrate obtained during the determination of the water-soluble matter according to ISO/R 787, Part III, and titrate with an approximately 0.01 N solution of sodium or potassium hydroxide, or hydrochloric or sulphuric acid by means of a microburette to a pH of 5.5, using a pH meter.

NOTE — If the filtrate is colourless, the determination may alternatively be carried out in accordance with ISO/R 787, Part IV.

6.3.2 *Expression of results*

Calculate the number of millilitres of 0.1 N solution per 100 g of pigment by the formula :

$$\frac{V \times T \times 2500}{m}$$

where

V is the volume, in millilitres, of 0.01 N solution used;

T is the normality of the 0.01 N solution;

m is the mass of the test portion, in grams, taken for the determination of the water-soluble matter content according to ISO/R 787, Part III.

Express the result in terms of the number of millilitres of 0.1 N solution required for 100 g of pigment.

6.4 Ease of dispersion

6.4.1 Carry out the test according to ISO/R 787, Part . . .[1], *Comparison of ease of dispersion of pigments (oscillatory shaking method)*, using an average milling concentration of 20 % (*m/m*) of pigment.

NOTE — If more than 30 min are required to obtain a fineness of 5 µm, the description of the test procedure shall be adjusted to involve milling to 10 µm only.

6.4.2 By agreement between purchaser and vendor, the test may alternatively be carried out with the automatic muller, according to ISO/R 787, Part . . .[1] *Comparison of ease of dispersion of pigments (automatic muller method).*

1) In preparation.