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Standard Specification for Micaceous Iron Oxide Pigments for Paint[1]

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1. Scope

1.1 This specification covers the requirements and methods of test for lamellar iron oxide pigments, commonly called micaceous iron oxide, used primarily in protective coatings for steel. This standard is related to ISO 10601 and Australian Standard 2855- covering both specifications and methods of test. A companion test method for lamellar (thin film) content is included. This specification covers two types of micaceous iron oxide.

1.2 The following hazard caveat pertains only to the test method portion, Section 9, of this specification: *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese[2]

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments[2]

D 1030 Test Method for Fiber Analysis of Paper and Paperboard[3]

D 1208 Test Methods for Common Properties of Certain Pigments[2]

2.2 *Other Standards:*

ISO-10601 Micaceous Iron Oxide Pigments for Paints—Specifications and Methods of Test[4]

2855-1986, Paint and Related Materials—Micaceous Iron Oxide Pigment[5]

2.3 ASTM Adjuncts:

Three photographs of two grades of micaceous iron oxide pigment[6]

3. Terminology

3.1 *Definitions:*

3.1.1 *lamellar particles*—thin flat particles.

3.1.2 *micaceous, lamellar*—similar to the structure of mica.

3.1.3 *micaceous or lamellar iron oxide (MIO)*— a refined mineral ore (also known as specular haemetite) or a manufactured product, consisting of iron (III) oxide (Fe_2O_3). It is grey in color with a metallic sheen and a lamellar form.

4. Significance and Use

4.1 The primary use of lamellar iron oxide is in protective coatings for steel and for optimum performance the pigment should have a high content of thin, flake-like particles. The protective action is ascribed to close packing of the pigment platelets within the paint film forming overlapping layers that lie roughly parallel to the substrate. This impedes penetration of corrosion stimulants, reduces ultraviolet degradation of the binder, improves film strength, reduces polymer swelling, and minimizes blistering. For less critical requirements, micaceous iron oxide with lower content of thin flakes may be acceptable. Therefore in this specification, micaceous iron oxide pigments are classified into two groups according to their thin flake content as determined by microscopic examination (see Section 9). When the proportion of lamellar particles is <50 %, the pigment is not to be classified as "lamellar" nor "micaceous". Information on MIO lamellarity and performance is detailed in JOCCA 64, JOCCA 66, March 1983, and JOCCA 66, June 1983.[7,8,9]

4.2 Micaceous iron oxide pigments can vary in composition, particle size range, and particle shape depending upon whether they are produced synthetically or, if a refined natural oxide, the location where the ore was mined. The lamellar ore is readily fractured into thin platelets. These thin platelets of hematite (Fe_2O_3) are structurally similar to mica, hence the term "micaceous". A hard granular form of the crystal also

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[2] *Annual Book of ASTM Standards*, Vol 06.03.

[3] *Annual Book of ASTM Standards*, Vol 15.09.

[4] Available from *International Organization for Standardization*, Case Postale 56, 11211 Geneve 20, Switzerland.

[5] Available from *Australian Standards Institute*, 80 Arthur St., North Sydney, N.S.W., Australia.

[6] Three photographs of two grades of micaceous iron oxide pigment are available at nominal cost from ASTM Headquarters. Order Adjunct ADJD5532.

[7] D.M. Bishop, "Micaceous Iron Oxide Pigments," *Journal of Oil and Colour Chemist's Association (JOCCA)*, Vol 64, February 1981.

[8] D.M. Bishop and F.G.R. Zobel, "Micaceous Iron Oxide Paints," *JOCCA*, Vol 66, March 1983.

[9] S. Wiktorek and J. John, "Micaceous Iron Oxide in Protective Coatings," *JOCCA*, Vol 66, June 1983.

exists and cannot be fractured into thin platelets. The granular form cannot readily form the overlapping shingle-like structure possible with the thin platelets.

5. Apparatus

5.1 *Glass Microscope Slide*, approximately 25 by 75 by 1 mm and cover slip.

5.2 *Glass Rod*, approximately 100-mm long, 5-mm diameter with rounded end.

5.3 *Optical Microscope*, capable of 200 × magnification with 20 × objective lens, fitted with high intensity substage illumination.

NOTE 1—Use of a "wide-field" eyepiece is recommended, preferably incorporating a frame-mask or graticule such that a rectangular frame image appears in the field of view.

6. Composition and Properties

6.1 The pigment shall conform to the requirements for properties prescribed as follows:

	Types	
	I, %	II, %
Iron content, expressed as iron III oxide (Fe_2O_3) (Test Methods D 50)	min 85	min 85
Volatile matter (Test Methods D 280)	max 0.5	max 0.5
Water solubility (Test Methods D 1208)	max 0.5	max 0.5
Lamellar content (see Section6)	>65	50 to 65
Maximum retained on 150-mesh (106-µm), U.S. standard seive No. 140	<0.1	<0.1

7. Sampling

7.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack or other unit of production in a shipment. When no markings distinguishing units of production appear, samples to be tested shall be taken from lot sizes agreed upon between the buyer and the producer. At the option of the purchaser, the samples may be tested separately or, after blending the samples from the same production unit in equal quantities, tested as a composite sample.

TEST FOR LAMELLAR CONTENT

8. Reagent

8.1 *Reagent*—refined linseed oil.

9. Procedure

9.1 Place a few milligrams of a representative sample of the dry pigment on the glass slide. Add a few drops of refined linseed oil and gently incorporate the pigment into the oil using the glass rod, spreading the mixture thinly and evenly. Place the cover slip over the pigment/oil dispersion and position on the microscope stage.

9.2 Spread a few milligrams of the sample of the dry pigment on the glass slide and examine it without adding any linseed oil as follows: Adjust the light intensity below the slide to the highest practical level. Focus sharply at 200 × magnification and scan the slide until a field of view is obtained which is considered to be representative and in which at least 50 particles can be seen.

9.3 Thin-flake lamellar iron-oxide particles will appear as sharply defined red translucent platelets while thicker particles will appear as black shapes. Platelets < 4 µm will have the red appearance and edges will appear fractured. Platelets of 4 to 10 µm thickness will have a darker red appearance with sufficient back light intensity. Particles thicker than 10 µm will appear black.

9.4 Assess the proportion of red to black particles and classify into the appropriate type as follows:

Type	Appearance	Thin Flakes, %
I	Proportion of red particles	>65
II	Proportion of red particles	50 to 65
Non-lamellar	Proportion of *black* particles	>50

9.5 If the thin flake content is not obvious, count the number of red and black particles in a group of at least 50 particles. A frame-image device (see Note 1) will assist this operation. Repeat the count on a duplicate slide and calculate the average percentage of red particles. To assist counting, it may be useful to use an eye pointer (see Test Method D 1030). Particles of a diameter less than 5 m are not to be counted.

9.6 Typical photographs[7] of the two grades of micaceous iron oxide pigment, viewed with an optical microscope by transmitted light are shown in Adjunct Figs. 1. Adjunct Fig. 3 illustrates > 50 % black granular pigment, classified non-lamellar or non-micaceous.

10. Report

10.1 The report shall contain at least the following information:

10.1.1 The identification of the product tested,

10.1.2 A reference to this specification,

10.1.3 The results of the tests and whether or not the production complies with the relevant specifications limit,

10.1.4 Any deviation from the procedures specified, and

10.1.5 The dates of the tests.

11. Precision and Bias

11.1 Precision and bias cannot be calculated because the results do not form a continuum and there is no reference material available.

12. Keywords

12.1 flake-like pigments; hematite; iron oxide; lamellar pigments; lamellar tests; micaceous iron oxide; MIO; MIO classification; permeability; synthetic MIO