

U. S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

CERTIFICATE

for

STANDARD SAMPLE 313

BLACK IRON OXIDE

Purpose of Standard.

This standard sample of black iron oxide has been established as a standard for Mass Color, Tinting Strength, and Character of Tint only, in connection with purchases of material made under Federal Specification TT-I-698, "Iron Oxide; Black, Synthetic, Dry (Paint Pigment)".

Properties of Standard.

This material is nearly pure oxide of iron ( $\text{Fe}_2\text{O}_3$ ). It is a manufactured product produced by precipitation, under controlled conditions, from an iron salt solution (copperas) by means of an alkali, with subsequent oxidation. When bottled, the sample was practically neutral, contained about 0.2 percent moisture, about 77 percent ferric oxide ( $\text{Fe}_2\text{O}_3$ ), and about 22 percent ferrous oxide ( $\text{FeO}$ ). It had a specific gravity of about 4.95. However, these values are not certified and are given only as matters of general information. A spectrophotometric record of the color of the standard is on file at this Bureau. The standard will be protected from dust, light, and moisture, and checked at intervals for color stability.

Methods of Test.

(a) Mass Color of Pigments.

The following is abstracted from Federal Specification TT-P-141b, Method 421.1, with some slight changes:

Transfer 1.000 g of the test sample and of the standard to a glass plate or stone slab, and rub up separately, using the same amount (0.50 ml is suggested) of the same raw linseed oil in each case. The raw linseed oil shall conform to Federal Specification TT-O-369. For control work, a burette is recommended for measuring the oil used, instead of weighing, because of the simplicity, speed, and accuracy obtainable with the burette. Allow sufficient time for the oil to drain to its true level. In case of doubt or dispute over any color, all portions shall be weighed. The weight of 0.50 ml of the specified raw linseed oil is about 0.467 g.

Mix the oil and pigment on the rubbing slab to a paste with a clean steel (not nickel-plated) spatula. When all of the dry pigment is worked up by means of the spatula, spread it over an area approximately 4 inches wide and 12 to 15 inches long. Rub up the paste with a glass muller, the grinding face of the muller to be 2 3/4 to 3 inches in diameter and kept sharp by lightly grinding with turpentine and No. 303 optical emery, or its equivalent.

In counting the rubs given a color, one stroke up and one stroke back is considered 1 rub. Allow the muller to travel up one side and back the other side, twisting the muller slightly at the top and bottom of each stroke to help work in the pigment. After each 30 rubs, "pick-up" the paste with the spatula by scraping the face of the muller and gathering the paste on the slab into a daub. Continue the mulling until the paste is given 150 rubs.

When the mulling is complete, place the sample and the reference standard in juxtaposition on a bright tin or clear glass panel. Make the daub of each about 1 inch wide and 2 inches long and draw a scraper lightly over the pastes to even off the ridges and to present both daubs on an even plane. Mass color shall be judged immediately. If glass is used, observe the rub-outs from the top and not through the glass panel.

(b) Tinting Strength and Character of Tint of Pigments.

The following is abstracted from Federal Specification TT-P-141b, Method 422.1, with some slight changes:

Weigh 0.1 g of the mulled color paste from the slab (see Method for Mass Color of Pigments) and counterbalance with the reference standard pigment paste. Then add 3.0 g of the "reduction paste" (zinc oxide paste in oil which conforms to Federal Specification TT-Z-301) to the sample and to the reference standard. Thoroughly mix each of the pastes on the flat glass or slab only until no more streaking is noticeable.

Place the sample and reference standard in juxtaposition on a bright tin or clear glass panel. Make the daub of each at least 1 inch wide and about 2 inches long and draw the scraper lightly over the pastes to even off the ridges and to present both daubs on an even plane. Judge the color immediately. If glass is used, observe the colors from the top and not through the glass panel.

If the sample differs from the standard in that it yields a different colored tint (for example, a brownish gray), it does not meet the specification in character of tint. If it shows the same character of tint and the rub-out is as dark as or darker than the standard, it meets the specification with regard to both character of tint and tinting strength.

Although intended to be used primarily as a color standard for dry pigment purchases, this standard sample may also be used (in the absence of a mutually agreed upon paste standard) in the purchase of color-in-oil pigments. Methods suggested for making the color comparisons are given below. Because of the necessarily arbitrary manner of preparing the standard pigment paste, it may be desirable to permit a small amount of latitude when making the color comparisons.

(c) Mass Color of Pastes.

Prepare a paste with the standard pigment in the same manner as for the determination of Mass Color of Pigments except that the percentage of pigment shall be the same as in the sample of paste being tested. Make the color comparison immediately in accordance with the procedure described in section (a).

(d) Tinting Strength and Character of Tint of Pastes.

Follow the same procedure as outlined for pigments except that approximately 0.1 g of the sample paste (containing volatile thinner) should be weighed first by difference, its exact weight recorded, and the same weight of paste prepared from the standard pigment and linseed oil then taken for comparison.

Recommendations.

This standard sample should be kept tightly covered and protected from dust, light, and moisture when not in use.

(Signed) LYMAN J. BRIGGS, Director.

Washington 25, D. C.

August 28, 1945.