



Designation: D 50 – 90 (Reapproved 1999)

Standard Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese¹

This standard is issued under the fixed designation D 50; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover procedures for the chemical analysis of yellow, orange, red, and brown pigments containing iron and manganese. The test methods apply specifically to the following pigments: synthetic hydrated yellow iron oxide, yellow ocher, red and brown iron oxides, raw and burnt umber, raw and burnt sienna, and venetian red.

1.2 The analytical procedures appear in the following order:

| | Sections | ASTM Method References |
|---------------------------------------|-----------|------------------------|
| Dry Pigments | | |
| Moisture and Other Volatile Matter | 6 | D 280 |
| Loss on Ignition | 7 | |
| Coarse Particles | 8 | D 185 |
| Matter Soluble in Water | 9 | D 1208 |
| Organic Coloring Matter | 10 | |
| Iron Oxide | 11 and 12 | |
| Calcium Compounds (Reported as CaO) | 13 and 14 | |
| Sulfates Soluble in Hydrochloric Acid | 15 and 16 | |
| Lead Chromate (in Ochres) | 17 | |
| Calcium Carbonate (in Venetian Red) | 18 | C 25 |
| Manganese (in Siennas and Umbers) | 19 and 20 | |
| Pigment Pastes in Oil | | |
| Pigment Content | 21 | D 1208 |
| Moisture and Other Volatile Matter | 22 | D 1208 |
| Nonvolatile Matter in Vehicle | 23 | |
| Moisture by Distillation | 24 | D 1208 |
| Coarse Particles and Skins | 25 | D 185 |
| Consistency (Stormer) | 26 | D 562 |

1.3 *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 appro-*

priate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- C 25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer⁴
- D 1193 Specification for Reagent Water⁵
- D 1208 Test Methods for Common Properties of Certain Pigments³
- E 11 Specification for Wire Cloth Sieves for Testing Purposes⁶

3. Significance and Use

3.1 These test methods compile in one place, recommended procedures for analyzing inorganic colored pigments. These pigments are used extensively in paints, and for this reason their compositions are important to the formulators and user.

4. Preparation of Sample

4.1 Mix the sample thoroughly and take a representative portion for analysis. Reduce any lumps or coarse particles to a fine powder by grinding. Grind extracted pigments to pass a No. 80 (180-μm) sieve (see Specification E 11 for detailed

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.31 on Pigment Specification.

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² *Annual Book of ASTM Standards*, Vol 04.01.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

⁵ *Annual Book of ASTM Standards*, Vol 11.01.

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

requirements). Discard any skins that do not pass through the sieve. Mix the finely ground pigment thoroughly.

5. Purity of Reagents

5.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁷ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent water conforming to Specification D 1193.

DRY PIGMENTS

Moisture and Other Volatile Matter

6. Procedure

6.1 Determine moisture and other volatile matter in accordance with Method A of Test Methods D 280.

Loss on Ignition

7. Procedure

7.1 Weigh accurately approximately 1 g of pigment into a previously weighed and ignited porcelain crucible (Note 1). Cover and ignite to constant weight over a bunsen or Meker burner or in an electric furnace at a temperature of approximately 900°C. Cool in a desiccator, weigh, and calculate the percent loss on ignition. This figure may include combined water, carbon dioxide (CO₂), organic matter, and some sulfuric oxide (SO₃) if much calcium sulfate (CaSO₄) is present. The CO₂ may be determined on a separate portion if desired.

NOTE 1—It is inadvisable to use platinum unless it is known that attacking substances are absent.

Coarse Particles

8. Procedure

8.1 Determine coarse particles in accordance with Test Method D 185.

Matter Soluble in Water

9. Procedure

9.1 Determine matter soluble in water in accordance with Test Methods D 1208.

Organic Coloring Matter

10. Procedure

10.1 Boil 2 g of the sample with 25 mL of water, let settle, and decant the supernatant liquid. Boil the residue with 25 mL of ethyl alcohol (95 %) and decant as before. Boil the residue

with 25 mL of 1 *N* alcoholic sodium hydroxide (NaOH) solution and again decant. Boil another 2-g portion of the sample with 25 mL of chloroform, let settle, and decant the supernatant liquid. If any one of the above solutions is colored, organic coloring matter is indicated (Note 2). If the solutions remain colorless, organic colors are probably absent, but may be tested for by reference to procedures given in standard reference works, taking into account the nature of the pigment involved.⁸

NOTE 2—With this class of pigments indication of presence of an organic color may often be noted by the characteristic odor given off on ignition.

Iron Oxide

11. Reagents

11.1 *Stannous Chloride Solution* (SnCl₂·2H₂O)—Dissolve 50 g of SnCl₂·2H₂O in 300 mL of hydrochloric acid (HCl) (sp gr 1.19) and dilute with water to 500 mL. Keep the clear solution in a tightly stoppered bottle containing some metallic tin.

11.2 *Mercuric Chloride Solution* (HgCl₂)—Prepare a saturated solution of HgCl₂ (60 to 100 g/L).

11.3 *Sulfuric-Phosphoric Acid Mixture*—Mix 150 mL of sulfuric acid (H₂SO₄) (sp gr 1.84) with 150 mL of phosphoric acid (H₃PO₄) (85 %) and dilute with water to 1 L.

11.4 *Diphenylamine Indicator*—Dissolve 1 g of diphenylamine in 100 mL of H₂SO₄ (sp gr 1.84).

11.5 *Standard Potassium Dichromate Solution* (K₂Cr₂O₇) (0.1 *N*)—Dissolve 4.904 g of K₂Cr₂O₇ in water and dilute to 1 L. Standardize against the National Bureau of Standards standard sample No. 27 of Sibley iron ore.

11.6 *Potassium Ferricyanide Solution* (K₃Fe(CN)₆)—A very dilute solution is most satisfactory. Dissolve approximately 0.01 g of K₃Fe(CN)₆ in 50 mL of water. This solution must be made fresh when wanted because it does not keep.

12. Procedure

12.1 According to the amount of iron in the pigment, weigh 0.3 to 1.0 g of the pigment and ignite in a porcelain crucible at a dull red heat to destroy organic matter. Transfer to a 400-mL beaker and add 25 mL of HCl (sp gr 1.19). Cover with a watchglass and digest just short of boiling (80 to 90°C) until no dark specks can be seen in the insoluble residue. The addition of a few drops of SnCl₂ solution after adding the acid greatly assists dissolving of the iron. When the residue is light in color, the solution of iron may be considered complete. This may take from 15 min to 1 h, or longer.

12.2 Add 25 to 50 mL of water and heat to gentle boiling (avoid vigorous prolonged boiling). Slowly add SnCl₂ solution dropwise until the last drop makes the solution colorless or free from any tinge of yellow, then add 1 or 2 drops in excess. It is best to keep the watchglass on the beaker while adding the SnCl₂ solution, with agitation of the hot iron solution after each addition. If too much SnCl₂ is added by mistake, add potassium

⁷ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

⁸ Reference may be made to the following: Payne, H. F., "Organic Coatings Technology," Vol II, John Wiley & Sons, Inc., New York, N. Y., 1961.