



Designation: D 444 – 88 (Reapproved 1999)

Standard Test Methods for Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow)¹

This standard is issued under the fixed designation D 444; 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 the pigment known commercially as “zinc yellow” or “zinc chromate yellow.”

1.2 The analytical procedures appear in the following order:

	Sections
Moisture and Other Volatile Matter	7
Combined Water	8
Chromium:	
Dichromate Method	9-11
Thiosulfate Method	9, 12, and 13
Zinc:	
Hydroxyquinoline Method	9, 14, and 15
Ferrocyanide Method	9, 16, and 17
Alkaline Salts	18 and 19
Sulfates	20 and 21
Chlorides	22 and 23
Matter Insoluble in Dilute Acetic Acid	24
Coarse Particles	25

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

2. Referenced Documents

2.1 ASTM Standards:

- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
- D 478 Specification for Zinc Yellow (Zinc Chromate)³
- D 1193 Specification for Reagent Water³

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 D 01.31 on Pigment Specifications.

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

³ *Annual Book of ASTM Standards*, Vol 11.01.

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁴

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁵

3. Significance and Use

3.1 This test method has been developed to standardize the chemical analysis of zinc chromate yellow pigment and to provide alternate methods of analysis for chromium and zinc.

4. Preparation of Sample

4.1 Mix the laboratory sample thoroughly. Take a sufficient quantity for the chemical analyses and pass it through a 180- μm (No. 80) sieve, grinding in a mortar if necessary.

NOTE 1—Detailed requirements for this sieve are given in Specification E 11.

5. 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.⁶ 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 for use in the preparation of reagents and in analytical procedures shall conform to Type II reagent water, in Specification D 1193.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ *Annual Book of ASTM Standards*, Vol 03.05.

⁶ *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.

6. Precision

6.1 Precision statements have not been established.

MOISTURE AND OTHER VOLATILE MATTER

7. Procedure

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

COMBINED WATER

8. Procedure

8.1 Weigh to 0.1 mg 3 g of the oven-dried material from the determination of moisture and other volatile matter (Note 2 and Note 3), and place in a porcelain boat. Introduce the boat with the charge into a refractory combustion tube in an electrically-heated combustion furnace of the type used for the determination of carbon in steel by direct combustion (Note 4). Place the boat at the center of the combustion tube maintained at a temperature of 1000°C for 4 h. Draw a current of pure dry air or dry nitrogen through the tube to sweep the evolved moisture into a previously weighed absorption tube containing anhydrous magnesium perchlorate ($Mg(ClO_4)_2$) or other efficient desiccant. The weight increase of the absorption tube represents the “combined water.”

NOTE 2—Loss on ignition of the pigment does not suffice for the determination of combined water in zinc yellow.

NOTE 3—If the pigment contains an organic treating agent, first remove this treating agent by washing with ether or chloroform.

NOTE 4—See Apparatus No. 1 for the determination of total carbon by direct combustion as described in Practices E 50.

SPECIMEN SOLUTION FOR DETERMINATION OF CHROMIUM AND ZINC

9. Procedure

9.1 Weigh to 0.1 mg about a 4-g specimen and mix with 50 mL of cold sulfuric acid (H_2SO_4) (1+5). It should dissolve completely at this stage (Note 5). Dilute the solution to 500 mL in a volumetric flask.

NOTE 5—A cloudy solution may result if the pigment contains a surface-treating agent. In this case, it usually can be cleared by cooling in an ice-bath and filtering through a medium porosity fritted disk. If the solution is not clarified by this treatment, extract a portion of the original sample with a solvent such as chloroform before the analysis is begun.

CHROMIUM BY THE DICHROMATE TEST METHOD

10. Reagents

10.1 *Ferrous Ammonium Sulfate Solution*—Dissolve 80 g of ferrous ammonium sulfate ($FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$) in 50 mL of H_2SO_4 (sp gr 1.84) and enough water to make 1 L of solution. Mix thoroughly before use. This solution is approximately 0.2 N.

10.2 *Ortho-Phenanthroline Indicator Solution*—0.5 % in water.

10.3 *Potassium Dichromate, Standard Solution* (0.1 N)—Dissolve 4.9035 g of dried potassium dichromate ($K_2Cr_2O_7$) in water and dilute to 1 L in a volumetric flask.

11. Procedure

11.1 Pipet 50 mL of the solution of the specimen (Section 7) into a 600-mL beaker. Add 200 to 250 mL of water and 10 mL of H_2SO_4 (sp gr 1.84). Cool to 35°C or below.

11.2 Add an excess of ferrous ammonium sulfate solution and back-titrate with 0.1 N $K_2Cr_2O_7$ solution, using ortho-phenanthroline indicator. Carry out a blank titration of the same amount of ferrous ammonium sulfate solution at the same time and in the same manner.

11.3 *Calculation*—Calculate the percent of chromium C as CrO_3 , as follows:

$$C = (B_1 - V_1)N_1 \times 0.03334/S_1 \times 100$$

where:

B_1 = $K_2Cr_2O_7$ solution required for titration of the blank, mL,

V_1 = $K_2Cr_2O_7$ solution required for back-titration of the specimen, mL,

N_1 = normality of the $K_2Cr_2O_7$ solution, and

S_1 = specimen in the aliquot used, g.

0.03334 = milliequivalents weight of CrO_3 .

11.4 Alternatively, the solution of the specimen may be titrated directly with the ferrous ammonium sulfate solution, using an electrometric titration assembly to detect the end point. Standardize the ferrous solution against $K_2Cr_2O_7$.

CHROMIUM BY THE THIOSULFATE TEST METHOD

12. Reagents

12.1 *Potassium Iodide Solution* (150 g/L)—Dissolve 150 g of potassium iodide (KI) in water and dilute to 1 L.

12.2 *Sodium Thiosulfate, Standard Solution* (0.1 N)—Dissolve 24.8 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in 1 L of freshly boiled and cooled water contained in a sterile glass bottle. If sulfur precipitates during preparation or upon subsequent use, discard the solution and prepare a new one. Standardize against iodine.

12.3 *Starch Indicator Solution*—Stir up 2 to 3 g of potato starch with 100 mL salicylic acid solution (1 %), and boil the mixture until the starch is practically dissolved, then dilute to 1 L with water.

13. Procedure

13.1 Pipet a 25-mL aliquot of the solution of the specimen (Section 9) into a 500-mL glass-stoppered Erlenmeyer iodimetric flask or other suitable glass-stoppered bottle containing 200 mL of H_2SO_4 (1+39). Add 20 mL of KI solution (150 g/L), stopper, and allow the solution to stand for approximately 5 min.

13.2 Titrate the liberated iodine with 0.1 N $Na_2S_2O_3$ solution at room temperature until the reddish brown iodine color becomes quite faint. Add 5 mL of starch solution and continue the titration until the final color change becomes pale green with no blue tinge. Titrate this final titration by swirling the flask at least three times after each addition of the $Na_2S_2O_3$ solution, being sure that there is no further color change, especially at the final stage of the titration. The green end point is definite and sharp.