This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D444 – 88 (Reapproved 2020)

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

This standard is issued under the fixed designation D444; 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 U.S. 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 The values stated in SI units are to be considered the standard. The values given in parentheses are for information only.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D185 Test Methods for Coarse Particles in Pigments

D280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments

D478 Specification for Zinc Yellow (Zinc Chromate) Pigments

D1193 Specification for Reagent Water

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

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 E11.

5. Reagents

5.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of

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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] 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 D1193.

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 Methods D280.

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 E50.

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 of ($Na_2S_2O_3 \cdot 5H_2O$) 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.

[3] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

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.

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

$$C = [(V_2N_2 \times 0.03334)/S_2] \times 100$$

where:

V_2 = $Na_2S_2O_3$ solution required for titration of the specimen, mL
N_2 = normality of the $Na_2S_2O_3$ solution, and
S_2 = specimen in the aliquot used, g.

ZINC BY THE HYDROXYQUINOLINE TEST METHOD

(Suitable if No Interfering Substances Are Present)

14. Reagents

14.1 *Acetone Solution of 8-Hydroxyquinoline (50 g/L)*—Dissolve 5 g of 8-hydroxyquinoline in 100 mL of acetone.

15. Procedure

15.1 Pipet 50 mL of the solution of the specimen (Section 9) into a 250-mL beaker and dilute to 100 mL with water. Add 5 to 10 g of ammonium chloride (NH_4Cl) and heat to boiling. Add a slight excess of ammonium hydroxide (NH_4OH) and let stand a few minutes to allow any precipitate to coagulate. Filter through an ashless, rapid paper into a 400-mL beaker and wash.

15.2 Heat the filtrate to boiling and add 5 mL of NH_4OH (sp gr 0.90). Add dropwise 10 mL of the acetone solution of 8-hydroxyquinoline (Note 6). Let stand 10 to 20 min and filter through a medium-porosity sintered-glass crucible. Wash well with water.

NOTE 6—The reagent is used in acetone solution rather than alcohol solution to eliminate the danger of reducing some of the chromate by alcohol. Avoid adding an excess of reagent and lengthy boiling after its addition. Ten millilitres of hydroxyquinoline (50 g/L) is sufficient for a normal zinc yellow. The solution can be tested for complete precipitation, but since the reagent itself is rather insoluble, the results may be misleading. The insoluble reagent will dissolve in a hot solution and also in an excess of alcohol or acetone, whereas the zinc oxyquinolate will not.

15.3 Dry the precipitate at 165°C for at least 2 h and weigh as zinc oxyquinolate.

15.4 *Calculation*—Calculate the percent zinc A as zinc oxide (ZnO), as follows:

$$A = [(P \times 0.2303)/S_3] \times 100$$

where:

P = zinc oxyquinolate, g, and
S_3 = specimen in aliquot used, g.
0.2303 = ZnO/zinc oxyquinolate = 81.38/353.37

ZINC BY THE FERROCYANIDE TEST METHOD

16. Reagents

16.1 *Methyl Orange Indicator Solution*—Dissolve 0.1 g of methyl orange in 100 mL of water.

16.2 *Potassium Ferrocyanide, Standard Solution*—Dissolve 22 g of potassium ferrocyanide ($K_4Fe(CN)_6 \cdot 3H_2O$) in water and dilute to 1 L. To standardize, transfer 0.2 g of metallic zinc or freshly ignited ZnO to a 400-mL beaker. Dissolve in 10 mL of hydrochloric acid (HCl, sp gr 1.19) and 20 mL of water. Drop in a small piece of litmus paper, add ammonium hydroxide (NH_4OH) until slightly alkaline, then add HCl until just acid, and then 3 mL more of HCl. Dilute to about 250 mL with hot water and heat nearly to boiling. Run in the $K_4Fe(CN)_6$ solution slowly from a buret, while stirring constantly, until a drop tested on a white porcelain plate with a drop of the uranyl indicator solution shows a brown tinge after standing 1 min. Do not allow the temperature of the solution to fall below 70°C during the titration. Run a blank using the same amounts of reagents and water as in the standardization. The standardization must be made under the same conditions of temperature, volume, and acidity as obtained when the specimen is titrated. Calculate the strength of the $K_4Fe(CN)_6$ solution in terms of grams of zinc as follows:

$$Z = W/(V_3 - B_2)$$

where:

Z = zinc equivalent of the $K_4Fe(CN)_6$ solution, g/mL,
W = zinc used (or equivalent to the ZnO used), g,
V_3 = $K_4Fe(CN)_6$ solution required for titration of the standard, g, and
B_2 = $K_4Fe(CN)_6$ solution required for titration of the blank, mL.

16.3 *Thymol Blue Indicator Solution (0.5 g/L)*—Dissolve 0.1 g of thymol blue indicator in 200-mL of methanol, ethanol, or isopropanol.

16.4 *Uranyl Acetate Indicator Solution (50 g/L)*—Dissolve 5 g of $UO_2(C_2H_3O_2)_2 \cdot H_2O$ in water made slightly acid with acetic acid and dilute to 100 mL.

17. Procedure

17.1 Pipet 50 mL of the solution of the specimen (Section 9) into a 400-mL beaker. Add 35 mL of H_2SO_4 (1+5) and 3 to 4 drops of thymol blue indicator solution. First add NH_4OH (sp gr 0.90) and finally NH_4OH (1+10) until the color of the

indicator changes to a salmon shade intermediate between pink and yellow. This gives a pH of about 2.4. Dilute the solution to 300 mL and heat to just under boiling. Pass in H_2S at a moderate rate for 40 min. Allow the precipitate to settle for 1 h and filter. Wash the filter ten times with water saturated with hydrogen sulfide gas (H_2S).

17.2 Dissolve the precipitate in hot HCl (1+3) and wash the filter paper well with hot water. Boil out the H_2S, neutralize to methyl orange with NH_4OH, and dilute to 300 mL. Add 2 g of NH_4Cl and 3 mL of HCl (sp gr 1.19), and heat to boiling.

17.3 Titrate the hot solution with $K_4Fe(CN)_6$ solution (16.2), using uranyl acetate as an external indicator on a spot plate or 1 to 2 drops of ferrous ammonium sulfate solution as an internal indicator.

17.4 *Calculation*—Calculate the percent zinc *A* as ZnO, as follows:

$$A = (V_4 Z / S_4) \times 100$$

where:

V_4 = $K_4Fe(CN)_6$ solution required for titration of the specimen, mL,
Z = ZnO equivalent of the $K_4Fe(CN)_6$ solution, g/mL, and
S_4 = specimen in the aliquot used, g.

ALKALINE SALTS

18. Reagents

18.1 *Gelatin Solution (0.2 g/L)*—Dissolve 0.2 g of low-ash gelatin in water and dilute to 1 L.

18.2 *Lead Acetate Solution (100 g/L)*—Dissolve 117 g of $Pb(C_2H_3O_2)_2 \cdot 3H_2O$ in water and dilute to 1 L.

19. Procedure

19.1 Dissolve exactly 1 g (Note 7) of a specimen in 10 mL of acetic acid (1+1) and add 25 mL of water. Heat until dissolved. Dilute to 250 mL and heat to boiling. Add 20 mL of lead acetate solution (100 g/L) and allow the precipitate to settle. Filter and wash the precipitate with hot water.

NOTE 7—The specimen should not be greater than 1 g because above 1 g the loss in alkali metals due to adsorption on the precipitate becomes excessive.

19.2 Saturate the filtrate with hydrogen sulfide (H_2S) for 40 min. Add 10 mL of gelatin solution (0.2 g/L) and stir vigorously. Filter and wash with H_2S water acidified with a few drops of H_2SO_4 (1+1).

19.3 Add 5 mL of H_2SO_4 (1+1) to the filtrate and boil to a volume of 50 mL. Transfer to a silica dish and evaporate to dryness. Ignite gently.

19.4 Leach the residue with hot distilled water, transferring the entire contents of the silica dish to a small beaker. Saturate with H_2S for about 15 min (Note 8). Add 10 mL of gelatin solution (0.2 g/L) and stir vigorously. Filter and wash the precipitate with H_2S water, catching the filtrate in a tared silica dish.

NOTE 8—A second treatment with H_2S is necessary because some of the heavy metals pass through to the alkali metal filtrate.

19.5 Add about 2 mL of H_2SO_4 (1+1) to the filtrate, evaporate to dryness, and again ignite gently. During the ignition process add small portions of solid ammonium carbonate ($(NH_4)_2CO_3$). Cool in a desiccator and weigh.

19.6 Test the residue for calcium. If present, determine the amount, calculate to calcium sulfate ($CaSO_4$), and deduct from the weight of the ignited residue.

19.7 *Calculation*—Calculate the percent of alkaline salts *D* as potassium oxide (K_2O), as follows:

$$D = [(R - W_2) \times 0.541 / S_5] \times 100$$

where:

R = ignited residue (see 19.5), g,
W_2 = $CaSO_4$ (if any) (see 19.6), g, and
S_5 = sample used, g.

SULFATES

20. Reagent

20.1 *Barium Chloride Solution (100 g/L)*—Dissolve 117 g $BaCl_2 \cdot H_2O$ in water and dilute to 1 L.

21. Procedure

21.1 For this determination take about 20 g of Type I pigment or about 5 g of Type II pigment as defined in Specification D478. Weigh the specimen into a 1-L beaker. Add 75 mL of HCl (sp gr 1.19). Boil the solution under a hood until the rapid evolution of chlorine has subsided. Add 5 mL of methyl alcohol and boil to a volume of about 35 mL (Note 9). Add 25 mL of HCl and continue boiling. Add 5 mL of methyl alcohol and again boil to a volume of about 35 mL.

NOTE 9—If the volume is reduced below 35 mL, a precipitate may form that will not dissolve upon dilution. It is imperative, however, that all the chromium be reduced.

21.2 Dilute the solution to 400 mL with water. If the solution is not clear at this point, filter it. Heat to boiling and add 50 mL of glacial acetic acid. Add 10 mL of $BaCl_2$ solution (100 g/L) dropwise, while stirring. Boil for 30 min and allow to stand overnight (Note 10). Filter through a tared Gooch crucible and wash with hot water.

NOTE 10—The acetic acid is necessary to minimize the tendency of sulfate to form an inner complex with the trivalent chromium. It is necessary that the solution stand overnight to ensure the precipitation of all the sulfate.

21.3 Dry the precipitate in an oven, and ignite at 900°C to constant weight. Weigh as barium sulfate ($BaSO_4$).

NOTE 11—If the wet precipitate has a yellow appearance, all the chromate was not previously reduced. The results will be high and the specimen should be discarded. Greater attention should then be given to the reduction of the chromium. A slight greenish color cannot be avoided but does not indicate a serious error.

21.4 *Calculation*—Calculate the percent sulfates *E* as sulfur trioxide (SO_3), as follows:

$$E = [(P_2 \times 0.343) / S_6] \times 100$$

where:

P_2 = $BaSO_4$, g, and

S_6 = sample used, g.

CHLORIDES

22. Reagent

22.1 *Silver Nitrate Solution (17 g/L)*—Dissolve 17.0 g of $AgNO_3$ in water and dilute to 1 L.

23. Procedure

23.1 Weigh a 10-g specimen into a 600-mL beaker. Add 200 mL of water and 50 mL of nitric acid (HNO_3) (2+3). Warm just enough to dissolve the specimen. Filter to remove insoluble material. Add a slight excess of $AgNO_3$ solution (17 g/L) (Note 12). Boil for 5 min and let stand for about 2 h.

NOTE 12—If care is exercised in adding but a slight excess of $AgNO_3$ solution, no silver chromate (Ag_2CrO_4) will form. About 10 to 15 mL of $AgNO_3$ solution (17 g/L) is usually not too much.

23.2 If no crystals of Ag_2CrO_4 are present, filter at room temperature through a tared, fine-porosity sintered-glass crucible, using suction. Wash the precipitate free of $AgNO_3$ with HNO_3 (1+99). To be sure that an excess of $AgNO_3$ was used, test the filtrate by adding a few drops of HCl (2+3). Dry the precipitate at 105 ± 2°C for 2 h, cool, and weigh as silver chloride (AgCl).

23.3 If crystals of Ag_2CrO_4 are present (Note 13), filter the solution through a fine-porosity sintered-glass crucible and discard the filtrate containing most of the chromium. Dissolve the AgCl precipitate by pouring 100 mL of hot ammonium hydroxide (NH_4OH) (1+5) slowly through the crucible while applying gentle suction. Catch the filtrate in a clean flask, taking care not to lose any of it. Wash with a few millilitres of HNO_3 (sp gr 1.42) and then with a little more NH_4OH (1+5).

NOTE 13—Silver chromate is difficult to dissolve completely by washing with dilute HNO_3; hence, in such cases, it is necessary to dissolve in NH_4OH and reprecipitate.

23.4 Transfer the solution to a beaker and make it faintly acid by adjusting with either HNO_3 (1+5) or NH_4OH (1+5) as required. Add a few drops of $AgNO_3$ solution (17 g/L) and boil for 5 min. Let stand at least 2 h in a dark place. Filter through a tared, fine-porosity sintered-glass crucible, using suction.

23.5 Wash the precipitate free of $AgNO_3$ with 1 % (by volume) HNO_3, and dry at 105 ± 2°C for 2 h. Cool and weigh as AgCl.

23.6 *Calculation*—Calculate the percent chlorides E as chlorine, as follows:

$$E = \left[(P_3 \times 0.247)/S_7\right] \times 100$$

where:

P_3 = AgCl, g,
S_7 = specimen used, g, and
0.247 = Cl/AgCl = 34.45/143.32.

MATTER INSOLUBLE IN DILUTE ACETIC ACID

24. Procedure

24.1 Weigh to 0.1 mg about a 10-g specimen (Note 14) and place in a 600-mL beaker. Add 300 mL of acetic acid (1+9). Heat the mixture to 80°C and maintain at 80 ± 5°C, while stirring, until nothing further dissolves. Filter while hot through a tared Gooch crucible. Wash the insoluble residue on the filter with hot water.

NOTE 14—If the pigment contains an organic treating agent, first remove this treating agent by washing with ether or chloroform.

24.2 Dry the crucible at 105 ± 2°C and weigh.

24.3 *Calculation*—Calculate the percent of matter insoluble in 10 % (by volume) acetic acid F as follows:

$$F = (R_1/S_8) \times 100$$

where:

R_1 = residue, g, and
S_8 = specimen used, g.

COARSE PARTICLES

25. Procedure

25.1 Determine the percent of coarse particles in the pigment as received, in accordance with Test Methods D185.

26. Keywords

26.1 pigments—zinc chromate; zinc chromate

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