Designation: D1845 − 86 (Reapproved 2014)

Standard Test Methods for Chemical Analysis of Strontium Chromate Pigment[1]

This standard is issued under the fixed designation D1845; 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 the chemical analysis of strontium chromate pigment.

1.2 The analytical procedures appear in the following order:

	Sections
Strontium by the Strontium Sulfate Method	7 to 10
Chromium by the Thiosulfate Method	11 to 14
Chloride Content	15
Sulfate Content	16
Moisture and Other Volatile Matter	17
Coarse Particles	18
Mass Color and Tinting Strength	19

1.3 This 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 the safety concerns 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:*[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
D387 Test Method for Color and Strength of Chromatic Pigments with a Mechanical Muller
D444 Test Methods for Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow)
D1193 Specification for Reagent Water

3. Significance and Use

3.1 These test methods may be used to confirm the stated strontium oxide and chromium oxide content of strontium chromate.

4. Apparatus

4.1 *Gooch crucible.*

4.2 *Electric Furnace,* capable of 800°C.

5. Purity of Reagents

5.1 Reagent grade chemicals shall be used in all tests. 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.[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 Unless otherwise indicated, references to water shall be understood to mean Type II of Specification D1193.

6. Preparation of Sample

6.1 Mix the sample thoroughly. Take a sufficient quantity for chemical analysis and pass it through a No. 325 (4-µm) sieve.

STRONTIUM BY THE STRONTIUM SULFATE METHOD

7. Reagents

7.1 *Acetic Acid (glacial).*

7.2 *Ammonium Hydroxide (1+3)*—Mix 1 volume of concentrated ammonium hydroxide (NH_4OH, sp gr 0.90) with 3 volumes of water.

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[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.

[3] *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.

7.3 *Ethyl Alcohol,* conforming to Formula No. 2B or No. 30 of the U.S. Bureau of Alcohol, Tobacco, and Firearms.

7.4 *Hydrochloric Acid (1+1)*—Mix equal volumes of concentrated hydrochloric acid (HCl, sp gr 1.19) and water.

7.5 *Sulfuric Acid (1+19)*—Mix 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) with 19 volumes of water.

8. Procedure

8.1 Weigh to 0.1 mg about 0.5 g of the sample into a 400-mL beaker. Add 40 mL of HCl (1+1) and heat the solution to dissolve the pigment. Add 40 mL of ethyl alcohol until the chromium is reduced, as this is indicated by a dark coloration of the solution. Add to the solution 100 mL of water and sufficient NH_4OH (1+3) to form a slight persistent precipitate.

8.2 Add HCl dropwise until the precipitate just redissolves (Note 1). Heat the solution to just under boiling and add 20 mL of H_2SO_4 (1+19). Add 100 mL of ethyl alcohol and allow the precipitate to settle for several hours or overnight.

NOTE 1—For a cleaner precipitate, add HCl until a pH of 2.0 is reached, instead of adding HCl dropwise until the precipitate is just dissolved. Then add 25 mL of acetic acid to minimize the tendency of the sulfate to form a complex with trivalent chromium. Finally, heat the solution, and add H_2SO_4 (1 + 19).

8.3 Filter the precipitate through a Gooch crucible that has been previously dried to constant weight. Wash the precipitate several times with a solution of equal volumes of ethyl alcohol, water, and H_2SO_4 (1+19). Dry the crucible in an oven and then ignite for ½ h at 800°C or until constant weight is attained (weight loss less than 0.1 mg). Cool and weigh the crucible.

9. Calculation

9.1 Calculate the percent of strontium oxide (SrO) as follows:

$$\text{SrO, \%} = [(P \times 0.56416)/S] \times 100$$

where:

P = $SrSO_4$ precipitate, g, and
S = pigment specimen, g.

$$0.56416 = \frac{\text{molecular weight of SrO}}{\text{molecular weight of SrSO}_4} = \frac{103.63}{163.70}$$

10. Precision

10.1 *Within Laboratory*—The usual difference between duplicate runs performed by the same analyst is approximately 0.2 % of the SrO content of the pigment tested.

10.2 *Between Laboratories*—The average difference between two determinations performed by different analysts in different laboratories is approximately 0.5 % of the SrO content of the pigment tested.

CHROMIUM BY THE THIOSULFATE METHOD

11. Reagents

11.1 *Hydrochloric Acid (1+5)*—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 5 volumes of water.

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

11.3 *Sodium Thiosulfate Standard Solution (0.1 N)*—Dissolve 24.8 g of reagent grade $Na_2S_2O_3 \cdot 5H_2O$ in recently boiled water and dilute to 1 L with additional recently boiled water. To standardize, pipet 25 mL of the standard potassium dichromate solution (exactly 0.1000 *N*) into a 250-mL Erlenmeyer flask. Add 100 mL of water, 15 mL of potassium iodide solution, and 15 mL of concentrated hydrochloric acid (sp gr 1.19). Titrate the liberated iodine with sodium thiosulfate solution until the reddish-brown color becomes quite faint. Add 5 mL of starch solution and continue the titration dropwise until the blue color changes to a pale green. Calculate the normality of the sodium thiosulfate solution as follows:

$$N = \frac{2.5000}{M}$$

where:

N = normality, and
M = sodium thiosulfate solution, g.

11.4 *Starch Indicator Solution*—Make a homogenous paste of 10 g of soluble starch in cold water. Add to this 1 L of boiling water, stir rapidly, and cool. Salicylic acid (1.25 g/L) may be added to preserve the indicator. If long storage is required, the solution should be kept in a refrigerator at 4 to 10°C (40 to 50°F). Prepare fresh indicator when the end point of the titration from blue to colorless or blue to light green fails to be sharp.

12. Procedure

12.1 Weigh accurately approximately 0.2 g of the sample into a 250-mL iodine flask. Add 50 mL of water and 50 mL of HCl (1 + 5); swirl the flask to complete solution. Add 20 mL of KI solution and allow the solution to stand for several minutes.

12.2 Titrate the liberated iodine with 0.1 *N* $Na_2S_2O_3$ solution until the reddish brown iodine color becomes faint. Add 1 mL of starch solution and continue the titration cautiously to the end point which is easily discernible when the color changes from blue to a light green with no blue tinge.

13. Calculation

13.1 Calculate the percent of chromium as chromic oxide (CrO_3) as follows:

$$\text{CrO}_3\text{, \%} = [(VN \times 0.03334)/S] \times 100$$

where:

V = $Na_2S_2O_3$ solution required for titration of specimen, mL,
N = normality of the $Na_2S_2O_3$ solution, and
S = sample used, g.

14. Precision and Bias

14.1 *Within Laboratories*—The usual difference between duplicate runs performed by the same analyst is approximately 0.3 % of the CrO_3 content of the pigment tested.

14.2 *Between Laboratories*—The average difference between two determinations performed by different analysts in different laboratories is approximately 0.5 % of the CrO_3 content of the pigment tested.

$$0.0334 = \frac{\text{molecular weight } CrO_2}{1000 \times 3\ (\text{volume})} = \frac{99.99}{1000 \times 3}$$
$$= \text{milliequivalent weight}$$

CHLORIDE CONTENT

15. Procedure

15.1 Determine the chloride content in accordance with Test Methods D444.

SULFATE CONTENT

16. Procedure

16.1 Determine the sulfate content in accordance with the Test Methods D444.

MOISTURE AND OTHER VOLATILE MATTER

17. Procedure

17.1 Determine moisture and other volatile matter in accordance with Method A of Test Methods D280.

COARSE PARTICLES

18. Procedure

18.1 Determine the percentage of coarse particles in the pigment as received in accordance with Test Methods D185.

MASS COLOR AND TINTING STRENGTH

19. Procedure

19.1 Determine mass color and tinting strength in accordance with Test Method D387.

20. Keywords

20.1 chromium; strontium chromate pigment; thiosulfate method

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