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Standard Test Methods for Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate and Chromium Oxide Green[1]

This standard is issued under the fixed designation D126; 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, and green pigments containing lead chromate and chromium oxide green.

1.2 The analytical procedures appear in the following order:

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1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety problems, 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.* Specific hazard statements are given in Note 3.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

D521 Test Methods for Chemical Analysis of Zinc Dust (Metallic Zinc Powder)

D1013 Test Method for Determining Total Nitrogen in Resins and Plastics (Withdrawn 2007)[3]

D1193 Specification for Reagent Water

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Summary of Test Methods

3.1 *Chrome Yellow, Chrome Orange, and Molybdate Orange:*

3.1.1 Organic colors and lakes are determined qualitatively by boiling the sample in water, then ethyl alcohol, and finally chloroform.

3.1.2 Moisture and other volatile matter are determined in accordance with Test Method A of Test Methods D280.

3.1.3 Matter soluble in water is determined by boiling in water and filtering.

3.1.4 Lead chromate is determined by dissolving the sample in dilute HCl, filtering and titrating potentiometrically with $FeSO_4$ solution after addition of $HClO_4$.

3.1.5 Total lead is determined by precipitation as lead sulfide solution with H_2SO_4 and final precipitation as lead sulfate.

3.1.6 Sulfate is determined by dissolving the sample in acetic acid, neutralizing with sodium carbonate, plus addition

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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] The last approved version of this historical standard is referenced on www.astm.org.

of HCl to an aliquot followed by addition of $BaCl_2$ to precipitate as barium sulfate.

3.1.7 Carbon dioxide is determined by evolution.

3.1.8 Molybdenum is determined by precipitation as the sulfide, solution in HNO_3 and H_2SO_4, addition of NH_4OH and H_2SO_4. The solution is reduced in a Jones reductor, collected under $Fe_2(SO_4)_3$ solution and titrated with $KMnO_4$ solution.

3.1.9 Extenders are either:

3.1.9.1 Calcium carbonate, calcium sulfate, magnesium carbonate or;

(a) The compounds in 3.1.9.1 are determined qualitatively by precipitation with ammonium solution.

(b) If chromium is present, it is reduced and the lead salts dissolved in dissolving solution. Hydroxides and hydrous oxides are precipitated by addition of HCl and NH_4OH and filtered. CaC_2O_4 is precipitated with calcium oxalate solution and filtered, ashed and weighed as CaO. Alternatively, the precipitate is dissolved in H_2SO_4 and titrated with $KMnO_4$. Magnesium is determined on the filtrate from calcium determination by precipitation as the phosphate with ammonium phosphate solution.

3.2 *Chromium Oxide Green:*

3.2.1 Organic colors and lakes are determined qualitatively by boiling the sample in water, then ethyl alcohol, and finally choloroform.

3.2.2 Moisture and other volatile matter are determined in accordance with Test Method A of Test Methods D280.

3.2.3 Matter soluble in water is determined by boiling in water and filtering.

3.2.4 Total chromium as chromium oxide is determined by dissolving the sample in dilute HCl, filtering and titrating potentiometrically with $FeSO_4$ solution after addition of $HClO_4$.

4. Significance and Use

4.1 These test methods are for analysis designed as an aid in quality of yellow, orange, and green pigments containing lead chromate and chromium oxide green. Some sections may be applicable to analysis of these pigments when extracted from whole paints.

5. Purity of Reagents and Water

5.1 *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.[4] 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 *Water*—Unless otherwise indicated, references to water for use in the preparation of reagents and in analytical procedures shall be understood to mean reagent water conforming to Type II of Specification D1193.

6. Preparation of Sample

6.1 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 (Note 1). Discard any skins that do not pass through the sieve. Thoroughly mix the finely ground pigment and preserve in stoppered and suitably identified bottles or containers.

NOTE 1—Detailed requirements for this sieve are given in Specification E11.

6.2 Moisten the weighed portions of extracted pigments with a small amount of suitable wetting agent (Note 1) before adding reagents for analysis.

NOTE 2—A 0.1 % solution of sodium dioctylsuccinosulfonate has been found satisfactory. (This material is sold under the trade name of Aerosol OT.) Wetting agents containing mineral salts, sulfates, or sulfonates which may be hydrolyzed to sulfates, should be avoided; the use of alcohol is also undesirable because of its tendency to reduce chromates.

NOTE 3—**Warning:** As the National Institute for Occupational Safety and Health has stated that hexavalent chromium compounds are hazardous to health, care should be exercised in preparation of the sample. The wearing of a respirator and rubber or synthetic gloves are recommended. If hexavalent chromium materials come in contact with the skin, wash thoroughly with soap and water.

CHROME YELLOW, CHROME ORANGE, AND MOLYBDATE ORANGE

(Primrose, Lemon, and Medium Yellows; Chrome Oranges; Lead Molybdate or Basic Lead Chromate; Molybdate Orange)

ORGANIC COLORS AND LAKES

7. Procedure

7.1 Boil 2 g of the sample 2 min with 25 mL of water, let settle, and decant the supernatant liquid. Similarly, boil the residue with 25 mL of ethyl alcohol (absolute or 95 %) and decant as before. Likewise boil with 25 mL of chloroform and again decant. If any one of the above solutions is colored, organic colors are present. If all solutions remain colorless, organic colors are presumably absent. The presence of organic colors resistant to the above reagents is unlikely, but may be tested for by reference to procedures given in standard reference works.[5]

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[4] *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.

[5] Reference may be made to the following: Payne, H. F., "Organic Coatings Technology," Vol II, John Wiley & Sons, Inc., New York, NY, 1961.

MOISTURE AND OTHER VOLATILE MATTER

8. Procedure

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

MATTER SOLUBLE IN WATER

9. Procedure

9.1 Place 2.5 g of the sample in a graduated 250-mL flask. Add 100 mL of water and boil for 5 min. Cool, dilute to exactly 250 mL, mix, and allow to settle. Filter the supernatant liquid through a dry paper and discard the first 20 mL. Evaporate 100 mL of the clear filtrate to dryness in a weighed dish, heat for 1 h at 105 to 110°C, cool, and weigh.

9.2 *Calculation*—Calculate the % of matter soluble in water as follows:

$$\text{Matter soluble in water, \%} = (R \times 2.5 \times 100)/S \qquad (1)$$

where:

R = weight of residue, and
S = specimen weight, g.

LEAD CHROMATE[6]

10. Reagents

10.1 *Dissolving Solution*—Saturate 1 L of water with NaCl. Filter. Add to the filtered solution 150 mL of water and 100 mL of HCl (sp gr 1.19).

10.2 *Ferrous Sulfate, Standard Solution (0.3 N)*—Dissolve 86 g of $FeSO_4 \cdot 7H_2O$ in 500 mL of water to which 30 mL of H_2SO_4 (sp gr 1.84) has been added with constant stirring. Dilute to 1 L and standardize not more than 6 h before use by potentiometric titration against 0.7-g portions of $K_2Cr_2O_7$.

11. Procedure

11.1 Dissolve 1 g of the sample in 150 mL of the dissolving solution. Agitate for 10 to 15 min, keeping the solution cold until dissolution is complete (Note 4). If dissolution is not complete, filter through fine grade filter paper and wash with three 10-mL portions of cold dissolving solution. Add 10 mL of $HClO_4$ (70 %), dilute to 250 mL, and titrate potentiometrically with $FeSO_4$ solution.

NOTE 4—Incomplete solution of the pigment is evidence of the possible presence of barium sulfate, silica, silicates, or other acid-insoluble extenders (see Section 18). Some chrome yellows may contain organic addition agents and will give a turbid solution at this point.

Newer chemically resistant-type lead chromate type pigments (silica encapsulated) cannot be decomposed by the procedures described in this method. Pigments of this type may require treatment with strong alkali hydroxide or hydrofluoric acid.

Also, if trivalent antimony has been used in manufacturing the product, pentavalent antimony may be present which would interfere in the determination of lead chromate.

11.2 Alternatively, the solution may be reduced by a known excess of $FeSO_4$ solution and back-titrated with $KMnO_4$ solution in the presence of $MnSO_4$, or excess KI may be added and the liberated iodine titrated with $Na_2S_2O_3$ solution, using starch indicator. The iodine liberation method is not applicable in the presence of molybdenum.

TOTAL LEAD[6]

12. Procedure

12.1 Dissolve 0.5 g of sample as described in Section 11. Add 5 mL of ethyl alcohol (95 % or absolute) and boil until the chromium is reduced, as indicated by a green color. Filter if any insoluble residue is present, retaining the filtrate and washings for the determination. Add NH_4OH (sp gr 0.90) to this solution until a faint precipitate begins to form; then add 5 mL of HCl (sp gr 1.19) slowly, dilute to 500 mL, and pass a rapid current of H_2S into the solution until precipitation is complete. Settle, filter, and wash with water containing H_2S.

12.2 Rinse the precipitate from the filter (Note 5) into a beaker containing 25 mL of HNO_3 (1+3) and boil until all PbS has dissolved. Add 10 mL of H_2SO_4 (1+1) and evaporate to strong fumes of SO_3. Cool and add 50 mL of water and 50 mL of ethyl alcohol (95 %) (Note 6). Let stand 1 h; then filter on a tarred Gooch crucible. Wash with ethyl alcohol (95 %), dry, ignite at 500 to 600°C, and weigh as $PbSO_4$.

NOTE 5—If a trace of sulfide remains on the paper, the stained portion of the paper may be separately treated with bromine water, the paper filtered off, and the filtrate added to the body of the solution.

NOTE 6—Any sulfur remaining from decomposition of the sulfides may be mechanically removed as a globule of solidified sulfur at this point.

SULFATE[6]

13. Reagents

13.1 *Barium Chloride Solution*—Dissolve 117 g of $BaCl_2 \cdot 2H_2O$ in water and dilute to 1 L.

13.2 *Dissolving Solution*—See 10.1.

13.3 *Sodium Carbonate Solution (saturated)*—Prepare a solution containing excess Na_2CO_3 at laboratory temperature, and free of SO_4. Decant the clear solution for use as required.

14. Procedure

14.1 Digest 1.25 g of the sample with 100 mL of dissolving solution at 100°C for 5 min. Add 25 mL of glacial acetic acid and 15 mL of ethyl alcohol and heat gently for 10 min to reduce chromium, as indicated by the green color of the solution. Cool. Neutralize with saturated Na_2CO_3 solution and add a slight excess. Transfer to a 250-mL volumetric flask, dilute to the mark with distilled water, and mix. Filter without washing through a dry filter paper, discarding the first 10 to 15 mL.

14.2 Take a 200-mL aliquot of the filtrate, neutralize with HCl (1+1), and add 10 mL excess. Heat to boiling and boil for 5 min. To the gently boiling solution, add 15 mL of $BaCl_2$ solution dropwise with constant stirring. Digest on a steam bath for 2 h. Filter through an ignited tarred Gooch crucible,

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[6] Sections 23 and 24 under "Calculation of Substances Other than Insoluble Lead Compounds" should be read carefully before proceeding with the analyses described in Sections 10 to 22.

wash with HCl (1+99), and finally with hot water. Dry at 105 to 110°C, ignite at 900°C, and weigh.

CARBON DIOXIDE[6]

15. Procedure

15.1 Determine CO_2 by the evolution method on 2.5 g of the sample, using dilute HNO_3 free of NO or NO_2 and absorbing the CO_2 in soda lime or in KOH solution.

MOLYBDENUM[6]

16. Reagents

16.1 *Ferric Sulfate Solution*—Dissolve 20 g of $Fe_2(SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H_2O$ in 200 mL of water to which has been added 50 mL of H_2SO_4 (sp gr 1.84) and 20 mL of H_3PO_4 (85 %), and dilute to 1 L.

16.2 *Jones Reductor*—The reductor shall contain at least a 35-cm column of amalgamated zinc, prepared by shaking 20 to 30-mesh zinc free of iron or carbon with $HgCl_2$ solution (20 g/L) in sufficient quantity to produce an amalgam containing 1 to 5 % of mercury, and supported by a suitable inert pad of asbestos, glass wool, or other inert material.

16.3 *Potassium Permanganate, Standard Solution (0.1 N)*—Dissolve 3.16 g of $KMnO_4$ in water and dilute to 1 L. Let stand 8 to 14 days, siphon off the clear solution (or filter through a medium porosity fritted disk), and standardize against the National Bureau of Standards standard sample No. 40 of sodium oxalate ($Na_2C_2O_4$) as follows: In a 400-mL beaker dissolve 0.2500 to 0.3000 g of the Bureau of Standards sodium oxalate in 250 mL of hot water (80 to 90°C) and add 15 mL of H_2SO_4 (1+1). Titrate at once with $KMnO_4$ solution, stirring the liquid vigorously and continuously. The $KMnO_4$ must not be added more rapidly than 10 to 15 mL/min, and the last 0.5 to 1 mL must be added dropwise with particular care to allow each drop to be fully decolorized before the next is introduced. The solution shall not be below 60°C by the time the end point has been reached. (More rapid cooling may be prevented by allowing the beaker to stand on a small asbestos-covered hot plate during the titration. The use of a small thermometer (non-mercury type) as a stirring rod is most convenient.) Keep the $KMnO_4$ solution in a glass-stoppered bottle painted black to keep out light, or in a brown glass bottle stored in a dark place.

17. Procedure

17.1 Dissolve 1 g of the sample as described in Section 11. Add 5 mL of ethyl alcohol (95 % or absolute) and boil until chromium is reduced. Filter if any insoluble residue is present, retaining the filtrate and washings. Add NH_4OH (sp gr 0.90) cautiously until a faint precipitate begins to form, then add 15 mL of H_2SO_4 (sp gr 1.84) and dilute to 300 mL. Heat to boiling, pass in a rapid stream of H_2S for 15 min, and dilute with 300 mL of hot water. Pass in H_2S for 10 min, boil for 3 min, and cool. Pass in H_2S for 10 min, and let stand at room temperature for 1 h. Filter and wash with H_2SO_4 (1+99) saturated with H_2S.

17.2 Rinse the sulfide precipitate into the original beaker and add 20 mL of HNO_3 (sp gr 1.42) and 5 mL of H_2SO_4 (sp gr 1.84) (see Note 5). Cover and heat to fumes. Cool, add 10 mL of HNO_3 (sp gr 1.42), and again fume. Repeat this operation if necessary until a light-colored solution is obtained. Wash the cover and inside of the beaker and fume again to remove all HNO_3. Dilute to 200 mL and add NH_4OH (1+4) until neutral; then add 10 mL of H_2SO_4 (sp gr 1.84).

17.3 Cool the solution and reduce by passing through a Jones reductor at a rate not exceeding 100 mL/min, collecting the effluent under 200 mL of $Fe_2(SO_4)_3$ solution. Titrate with $KMnO_4$ solution. A blank determination should also be made.

EXTENDERS[6]

18. General Considerations

18.1 Extenders fall into two groups, depending on their solubility or insolubility in the dissolving solution described in Section 10, as follows:

A. Extenders Soluble in Dissolving Solution—Calcium sulfate (gypsum), calcium carbonate (whiting), and magnesium carbonate.

B. Extenders Insoluble in Dissolving Solution—Silica, magnesium silicate, and clay (Note 7).

18.2 Extenders of group *A* may be present if the analysis shows sulfates and carbonates to be in the pigment, and are absent if sulfate and carbonate are absent. Since the latter situation rarely exists, it is advisable to test for the presence of calcium and magnesium to determine if extenders are present. Extenders of group *B* are recognized as an insoluble residue following acid solution of the pigment, and may be determined quantitatively if desired, by the method described in Section 31. Extenders of group *A*, if present, may affect the calculation of insoluble lead compounds as given in Section 23. Their qualitative or quantitative estimation may be necessary.

NOTE 7—Some lead chromates may contain zirconium or titanium compounds, some of which are insoluble in the dissolving solution, but are not to be considered as extenders, since they have been added to improve the properties of the pigment.

Qualitative Detection of Extenders of Group A

19. Reagents

19.1 *Ammonium Phosphate Solution*—Dissolve 100 g of $(NH_4)_2HPO_4$ in water and dilute to 1 L.

20. Procedure

20.1 Dissolve 1 g of the sample as described in Section 11. Add 5 mL of ethyl alcohol (95 % or absolute) and boil until the chromium is reduced. An insoluble residue at this point denotes the presence of extenders of Group B. Filter if necessary and wash well.

20.2 To the filtrate, add NH_4OH (1+4) until just ammoniacal, boil 5 min, and allow to digest in a warm place until the precipitate has coagulated. Filter, washing well with hot water and reserving the filtrate. Dissolve the precipitate on the filter with HCl (1+1), washing back into the original beaker. Reprecipitate, filter, and wash as before.

20.3 Combine the washings, make just acid with HCl (1+1), and evaporate to a volume of about 250 mL. Add 50 mL of $(NH_4)_2HPO_4$ solution, cool, and add 50 mL of NH_4OH (sp gr 0.90). Allow to stand overnight. A precipitate indicates the presence of extenders of Group A.

Quantitative Determination of Extenders of Group A

21. Reagents

21.1 *Ammonium Oxalate Solution*—Dissolve 30 g of ammonium oxalate in water and dilute to 1 L.

21.2 *Ammonium Phosphate Solution*—See Section 19.

22. Procedure

22.1 If the sample is a chrome yellow or orange use the procedure given in Section 14 for dissolving and reducing chromium. For other chromium pigments heat gently 2 g of sample in a porcelain dish without ignition until iron blue, if present, is just decomposed. Transfer to a beaker and dissolve the lead salts in 150 mL of dissolving solution as described in Section 11.

22.2 Add 20 ml of HCl (sp gr 1.19) and digest 1 h at 100°C. Dilute to 300 mL, filter and wash thoroughly. Add NH_4OH (1+4) to the filtrate and washings until just ammoniacal, boil 5 min and allow to digest in a warm place until the hydroxides and hydrous oxides are coagulated. Filter, washing well with diluted water and reserve the filtrate. Redissolve the precipitate with HCl (1+1) washing back into the original beaker. Reprecipitate, filter and wash as before. Combine the washings with the original filtrate and add 50 mL of ammonium oxalate solution. Filter off the CaC_2O_4 precipitate on quantitative paper. Transfer to a tarred crucible, ash, ignite at 1300°C, cool in a desiccator, and weigh as CaO. Alternatively, the washed precipitate may be dissolved in H_2SO_4 (1+1) and the resulting solution titrated hot with 0.1 N $KMnO_4$ solution, as described in the Procedure section under Calcium in Test Methods D521.

22.3 Make the filtrate from the calcium determination slightly acid with HCl (1+1) and evaporate to 250 mL volume. Add 50 mL of $(NH_4)_2HPO_4$ solution, cool, and add 50 mL of NH_4OH (sp gr 0.90). Allow to stand overnight. Filter on suitable quantitative paper, wash with NH_4OH (1+19), ignite in a platinum crucible at 1050°C for 1 h and weigh. Care must be taken to char the paper slowly before igniting.

22.4 *Calculation*—Calculate the percent of extenders (as oxides) as follows:

$$\text{Calcium oxide, \%} = \text{weight of CaO} \times 50 \tag{2}$$

$$\text{Magnesium oxide, \%} = \text{weight of } Mg_2P_2O_7 \times 18.11 \tag{3}$$

The calculation of the amount of extenders as carbonates or sulfates is described in Section 24.

CALCULATION OF SUBSTANCES OTHER THAN INSOLUBLE LEAD COMPOUNDS

23. Calculations Where Extenders Are Absent

23.1 "Insoluble lead compounds" in chrome yellow and chrome orange may consist of $PbCrO_4$, $PbSO_4$, $2PbCO_3 \cdot Pb(OH)_2$, PbO, or $PbMoO_4$. The first two are characteristic of the chrome yellows, the first, third, and fourth of the basic chrome oranges, and the first, second, and last of molybdate oranges. For purpose of determining conformance with specification requirements, where "insoluble lead compounds" are defined as above, make the following calculations if extenders are absent:

23.2 If molybdenum is present, calculate the percent of $PbMoO_4$ from the titration of Section 17 as follows:

$$A = PbMoO_4, \% = \text{mL titration} \times \text{normality of } KMnO_4 \times 12.24 \tag{4}$$

23.3 Calculate the percent of $PbCrO_4$ from the titration of Section 11 as follows:

$$B = PbCrO_4, \% = \text{mL titration} \times \text{normality of } FeSO_4 \times 10.77 \tag{5}$$

23.4 Calculate the percent of total lead as oxide and the percent of excess PbO from the analysis of Section 12 as follows:

$$C = PbO, \% = \text{grams of } PbSO_4 \times 147.2 \tag{6}$$

$$D = \text{excess PbO}, \% = C - (0.6906\,B + 0.6078\,A) \tag{7}$$

23.5 Calculate the sulfate as SO_3 from the analysis of Section 14 as follows:

$$E = SO_3, \% = \text{grams of } BaSO_4 \times 34.3 \tag{8}$$

23.5.1 If E is equal to or greater than 0.3587 D, the % of $PbSO_4$ equals 1.3587 D, and the % total insoluble lead compounds equals $A + B + 1.3587\,D$.

23.5.2 If E is less than 0.3587 D, the percentage of $PbSO_4$ equals 3.788 E, and a new excess of PbO is calculated as follows:

$$F = \text{Excess PbO}, \% = D - 2.788\,E \tag{9}$$

23.6 Calculate the percent of CO_2 from the analysis of Section 15 as follows:

$$G = CO_2, \% = \text{grams of } CO_2 \times 40 \tag{10}$$

23.6.1 If G is equal to or greater than 0.1314 F, the percent of basic lead carbonate ($2\,PbCO_3 \cdot Pb(OH)_2$) is 1.1584 F and the percent of total insoluble lead compounds is $A + B + 3.788\,E + 1.1584\,F$.

23.6.2 If G is appreciably in excess, extenders are probably present. If G is less than 0.1314 F, the percent of $2\,PbCO_3{\cdot}Pb(OH)_2$ is 8.813 G and the excess PbO is:

$$H = \text{Excess PbO}, \% = F - 7.608\,G \tag{11}$$

23.6.2.1 The percent of total insoluble lead compounds is $A + B + 3.788\,E + 8.813\,G + H$.

24. Calculations Where Extenders Are Present

24.1 If extenders are present, calculate the CO_2 equivalent as follows:

$$I = CO_2 \text{ equivalent of extenders} \tag{12}$$

$$= \text{CaO}, \% \times 0.786 + \text{MgO}, \% \times 1.100$$

24.2 If I is less than G, use $G - I$ as the net CO_2 for purposes calculating the $2PbCO_3{\cdot}Pb(OH)_2$ content of the pigment.

$$\text{Total extenders} = \text{CaO}, \% + \text{MgO}, \% + I \tag{13}$$

24.3 If I is greater than G, some of the extender is present as $CaSO_4$ (Note 8). Calculate the total percent of extenders as follows:

$$J = CO_2 \text{ equivalent of MgO} = \text{MgO } \% \times 1.100 \qquad (14)$$

$$K = CO_2 \text{ present as } CaCO_3 = G - J \qquad (15)$$

$$CaCO_3, \% = 2.73\ K \qquad (16)$$

$$L = \text{CaO present as sulfate, } \% = \text{CaO, } \% \pm 1.27\ K \qquad (17)$$

$$CaSO_4, \% = 2.43\ L \qquad (18)$$

$$SO_3 \text{ combined as } CaSO_4, \% = 1.43\ L \qquad (19)$$

24.3.1 $E - 1.43\ L$ equals the net SO_3 to be used for calculating the $PbSO_4$ content of the pigment. If I is greater than G, no calculation of $2PbCO_3 \cdot Pb(OH)_2$ is necessary, and the excess PbO is given by F.

$$\text{Total extenders, } \% = 2.100 \times \text{MgO, } \% + 2.73\ K + 2.43\ L \qquad (20)$$

NOTE 8—It is impractical to detect by chemical means whether a pigment contains $CaCO_3$ and $PbSO_4$, or $CaSO_4$ and $2PbCO_3 \cdot Pb(OH)_2$. This calculation assumes the former as more probable.

PURE CHROME GREEN AND REDUCED CHROME GREEN

ORGANIC COLORS AND LAKES

25. Procedure

25.1 Determine organic colors and lakes in accordance with Section 7.

MOISTURE AND OTHER VOLATILE MATTER

26. Procedure

26.1 Determine moisture and other volatile matter in accordance with Section 8.

MATTER SOLUBLE IN WATER

27. Procedure

27.1 Determine matter soluble in water in accordance with Section 9.

IRON BLUE

28. Procedure

28.1 Determine the total nitrogen (by the Kjeldahl-Gunning method) (Note 9) on 1 g of the sample, adding 2 g of $FeSO_4 \cdot 7H_2O$ before digestion for at least 2½ h.

NOTE 9—For further details refer to Test Method D1013.

28.2 *Calculation*—Calculate the percent of iron blue as follows:

$$\text{Iron blue, } \% = \text{nitrogen, } \% \times 3.4 \qquad (21)$$

NOTE 10—Qualitative tests may be made for other nitrogen-containing blue pigments.

LEAD CHROMATE

29. Reagents

29.1 *Ferrous Sulfate, Standard Solution (0.3 N)*—See 10.2.

29.2 *Potassium Permanganate Standard Solution (0.1 N)*—Prepare as described in 16.3. Standardization is unnecessary except for the alternative procedure given in Section 30.

30. Procedure

30.1 Mix thoroughly 1 g of sample with at least 10 g of Na_2O_2 in a 30-mL pure iron crucible. Heat gently until the fusion is complete; then heat strongly, rotating the crucible with iron tongs (not Nichrome or chromium plate) for a few minutes to ensure complete fusion. Allow to cool; then transfer the crucible and cover to a beaker containing 250 mL of water. When the action has ceased, rinse and remove the crucible and cover, and boil for at least 10 min to destroy excess peroxide. Make just acid with H_2SO_4 (1+1) and add about 35 mL in excess. Add 2.5 mL of 0.1 N $KMnO_4$ solution and heat to boiling. Add 10 mL of HCl (1+4) and boil to reduce manganese and lead as indicated by clearing of the solution. If the solution is not clear after 5 min boiling, add 5 mL of additional HCl (1+4), and repeat until a clear solution is obtained. Cool and titrate with $FeSO_4$ solution on a potentiometric apparatus; or alternatively, add a measured excess of $FeSO_4$ and back-titrate with $KMnO_4$ solution in the presence of $MnSO_4$.

BARIUM SULFATE AND INSOLUBLE SILICEOUS MATERIAL[7]

31. Procedure

31.1 Heat gently 1 g of sample in a porcelain dish without ignition until the iron blue is just decomposed. Transfer to a beaker and dissolve the lead salts as described in Section 11, warming if necessary. Filter through a tared Gooch crucible, wash thoroughly, ignite at 600 to 800°C, and weigh.

31.2 *Calculation*—Calculate the percent of $BaSO_4$ and insoluble siliceous material as follows:

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[7] See Note 7 of Section 18.

$$\text{BaSO}_4 \text{ and insoluble siliceous material, \%} = \text{weight of precipitate} \times 100 \quad (22)$$

TOTAL LEAD

32. Procedure

32.1 Determine total lead on the filtrate from the determination of $BaSO_4$ and insoluble matter (Section 31) in accordance with the directions of Section 12.

SULFATE

33. Procedure

33.1 After decomposition of the iron blue in 1.25 g of sample as described in Section 31, digest the residue with 20 mL of HCl (sp gr 1.19) at 100°C until solution is complete. Add 300 mL of water and heat to boiling. Filter and wash thoroughly. Determine sulfate on the filtrate and washings as described in Section 14.

CALCIUM OXIDE SOLUBLE IN ACID

34. Reagents

34.1 *Ammonium Oxalate Solution*—See 21.1.

35. Procedure

35.1 Decompose 2 g of sample as described in Section 31. Add 20 mL of HCl (sp gr 1.19) and proceed in accordance with 21.1 to the end of the calcium determination.

35.2 *Calculation*—Calculate the percent of CaO soluble in acid as follows:

$$\text{CaO soluble in acid, \%} = \text{grams CaO} \times 50 \quad (23)$$

EXTENDERS

36. Procedure

36.1 The method described in Sections 31, 34, and 35 suffices for the detection and estimation of all common extenders except $MgCO_3$. This may be tested for the filtrate from the CaO determination by the method described in Section 22.

CALCULATION OF INSOLUBLE LEAD COMPOUNDS

37. Procedure

37.1 The insoluble lead compounds may be calculated by the methods of Sections 23 and 24.

CHROMIUM OXIDE GREEN

ORGANIC COLORS AND LAKES

38. Procedure

38.1 Determine organic colors and lakes in accordance with Section 7.

MOISTURE AND OTHER VOLATILE MATTER

39. Procedure

39.1 Determine in accordance with Section 8.

MATTER SOLUBLE IN WATER

40. Procedure

40.1 Determine in accordance with Section 9.

TOTAL CHROMIUM AS CHROMIUM OXIDE

41. Procedure

41.1 Using 0.4 g of the sample proceed as in Section 30.

41.2 *Calculation*—Calculate the percent of chromium as Cr_2O_3 as follows:

$$\text{Cr}_2\text{O}_3\text{, \%} = \text{mL titration} \times \text{normality of FeSO}_4 \times 63.34 \quad (24)$$

42. Precision and Bias

42.1 Data are not available to determine the precision and bias of these test methods. There are no plans at present to obtain such data.

43. Keywords

43.1 chromium oxide green pigment; green pigment containing lead chromate and chromium oxide; lead chromate pigment; lead containing pigment; molybdate pigment; orange pigment containing lead chromate and chromium oxide; yellow pigment containing lead chromate and chromium oxide

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