



Designation: D126 – 87 (Reapproved 2023)

# Standard Test Methods for Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate and Chromium Oxide Green<sup>1</sup>

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 ( $\epsilon$ ) 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 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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CHROME YELLOW, CHROME ORANGE, AND MOLYBDATE ORANGE	
Organic Colors and Lakes	7
Moisture and Other Volatile Matter	8
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Total Chromium as Chromium Oxide	41

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Note 3.*

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:<sup>2</sup>

- 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- D1013 Test Method for Determining Total Nitrogen in Resins and Plastics (Withdrawn 2007)<sup>3</sup>
- 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.

<sup>1</sup> 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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<sup>2</sup> 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.

<sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

3.1.4 Lead chromate is determined by dissolving the sample in dilute HCl, filtering and titrating potentiometrically with FeSO<sub>4</sub> solution after addition of HClO<sub>4</sub>.

3.1.5 Total lead is determined by precipitation as lead sulfide solution with H<sub>2</sub>SO<sub>4</sub> 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 of HCl to an aliquot followed by addition of BaCl<sub>2</sub> 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<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub>, addition of NH<sub>4</sub>OH and H<sub>2</sub>SO<sub>4</sub>. The solution is reduced in a Jones reductor, collected under Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> solution and titrated with KMnO<sub>4</sub> 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<sub>4</sub>OH and filtered. CaC<sub>2</sub>O<sub>4</sub> is precipitated with calcium oxalate solution and filtered, ashed and weighed as CaO. Alternatively, the precipitate is dissolved in H<sub>2</sub>SO<sub>4</sub> and titrated with KMnO<sub>4</sub>. 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 chloroform.

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<sub>4</sub> solution after addition of HClO<sub>4</sub>.

#### 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.

### 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

#### 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.<sup>4</sup> 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.

<sup>4</sup> *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.

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.<sup>5</sup>

## 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 °C to 110 °C, cool, and weigh.

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

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

where:

$R$  = weight of residue, and

$S$  = specimen weight, g.

## LEAD CHROMATE<sup>6</sup>

### 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  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  in 500 mL of water to which 30 mL of  $\text{H}_2\text{SO}_4$  (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  $\text{K}_2\text{Cr}_2\text{O}_7$ .

### 11. Procedure

11.1 Dissolve 1 g of the sample in 150 mL of the dissolving solution. Agitate for 10 min 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  $\text{HClO}_4$  (70 %), dilute to 250 mL, and titrate potentiometrically with  $\text{FeSO}_4$  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

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

<sup>6</sup> 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.

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  $\text{FeSO}_4$  solution and back-titrated with  $\text{KMnO}_4$  solution in the presence of  $\text{MnSO}_4$ , or excess KI may be added and the liberated iodine titrated with  $\text{Na}_2\text{S}_2\text{O}_3$  solution, using starch indicator. The iodine liberation method is not applicable in the presence of molybdenum.

## TOTAL LEAD<sup>6</sup>

### 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  $\text{NH}_4\text{OH}$  (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  $\text{H}_2\text{S}$  into the solution until precipitation is complete. Settle, filter, and wash with water containing  $\text{H}_2\text{S}$ .

12.2 Rinse the precipitate from the filter (**Note 5**) into a beaker containing 25 mL of  $\text{HNO}_3$  (1+3) and boil until all PbS has dissolved. Add 10 mL of  $\text{H}_2\text{SO}_4$  (1+1) and evaporate to strong fumes of  $\text{SO}_3$ . 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 °C to 600 °C, and weigh as  $\text{PbSO}_4$ .

**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<sup>6</sup>

### 13. Reagents

13.1 *Barium Chloride Solution*—Dissolve 117 g of  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  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  $\text{Na}_2\text{CO}_3$  at laboratory temperature, and free of  $\text{SO}_4$ . 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  $\text{Na}_2\text{CO}_3$  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 mL 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<sub>2</sub> solution dropwise with constant stirring. Digest on a steam bath for 2 h. Filter through an ignited tarred Gooch crucible, wash with HCl (1+99), and finally with hot water. Dry at 105 °C to 110 °C, ignite at 900 °C, and weigh.

### CARBON DIOXIDE<sup>6</sup>

#### 15. Procedure

15.1 Determine CO<sub>2</sub> by the evolution method on 2.5 g of the sample, using dilute HNO<sub>3</sub> free of NO or NO<sub>2</sub> and absorbing the CO<sub>2</sub> in soda lime or in KOH solution.

### MOLYBDENUM<sup>6</sup>

#### 16. Reagents

16.1 *Ferric Sulfate Solution*—Dissolve 20 g of Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> · (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> · 24H<sub>2</sub>O in 200 mL of water to which has been added 50 mL of H<sub>2</sub>SO<sub>4</sub> (sp gr 1.84) and 20 mL of H<sub>3</sub>PO<sub>4</sub> (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<sub>2</sub> 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<sub>4</sub> in water and dilute to 1 L. Let stand 8 days 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<sub>2</sub>C<sub>2</sub>O<sub>4</sub>) as follows: In a 400 mL beaker dissolve 0.2500 g to 0.3000 g of the Bureau of Standards sodium oxalate in 250 mL of hot water (80 °C to 90 °C) and add 15 mL of H<sub>2</sub>SO<sub>4</sub> (1+1). Titrate at once with KMnO<sub>4</sub> solution, stirring the liquid vigorously and continuously. The KMnO<sub>4</sub> must not be added more rapidly than 10 mL/min to 15 mL/min, and the last 0.5 mL 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<sub>4</sub> 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<sub>4</sub>OH (sp gr 0.90) cautiously until a faint precipitate begins to form, then add 15 mL of H<sub>2</sub>SO<sub>4</sub> (sp gr 1.84) and dilute to 300 mL. Heat to boiling, pass in a rapid stream of H<sub>2</sub>S for 15 min, and dilute with 300 mL of hot water. Pass in H<sub>2</sub>S for 10 min, boil for

3 min, and cool. Pass in H<sub>2</sub>S for 10 min, and let stand at room temperature for 1 h. Filter and wash with H<sub>2</sub>SO<sub>4</sub> (1+99) saturated with H<sub>2</sub>S.

17.2 Rinse the sulfide precipitate into the original beaker and add 20 mL of HNO<sub>3</sub> (sp gr 1.42) and 5 mL of H<sub>2</sub>SO<sub>4</sub> (sp gr 1.84) (see Note 5). Cover and heat to fumes. Cool, add 10 mL of HNO<sub>3</sub> (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<sub>3</sub>. Dilute to 200 mL and add NH<sub>4</sub>OH (1+4) until neutral; then add 10 mL of H<sub>2</sub>SO<sub>4</sub> (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<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> solution. Titrate with KMnO<sub>4</sub> solution. A blank determination should also be made.

### EXTENDERS<sup>6</sup>

#### 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<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> 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<sub>4</sub>OH (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  $(\text{NH}_4)_2\text{HPO}_4$  solution, cool, and add 50 mL of  $\text{NH}_4\text{OH}$  (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  $\text{NH}_4\text{OH}$  (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  $\text{CaC}_2\text{O}_4$  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  $\text{H}_2\text{SO}_4$  (1+1) and the resulting solution titrated hot with 0.1 N  $\text{KMnO}_4$  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  $(\text{NH}_4)_2\text{HPO}_4$  solution, cool, and add 50 mL of  $\text{NH}_4\text{OH}$  (sp gr 0.90). Allow to stand overnight. Filter on suitable quantitative paper, wash with  $\text{NH}_4\text{OH}$  (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:

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

$$\text{Magnesium oxide, \%} = \text{weight of Mg}_2\text{P}_2\text{O}_7 \times 18.11 \quad (3)$$

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  $\text{PbCrO}_4$ ,  $\text{PbSO}_4$ ,  $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$ ,  $\text{PbO}$ , or  $\text{PbMoO}_4$ . 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  $\text{PbMoO}_4$  from the titration of Section 17 as follows:

$$A = \text{PbMoO}_4, \% = \text{mL titration} \times \text{normality of KMnO}_4 \times 12.24 \quad (4)$$

23.3 Calculate the percent of  $\text{PbCrO}_4$  from the titration of Section 11 as follows:

$$B = \text{PbCrO}_4, \% = \text{mL titration} \times \text{normality of FeSO}_4 \times 10.77 \quad (5)$$

23.4 Calculate the percent of total lead as oxide and the percent of excess  $\text{PbO}$  from the analysis of Section 12 as follows:

$$C = \text{PbO, \%} = \text{grams of PbSO}_4 \times 147.2 \quad (6)$$

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

23.5 Calculate the sulfate as  $\text{SO}_3$  from the analysis of Section 14 as follows:

$$E = \text{SO}_3, \% = \text{grams of BaSO}_4 \times 34.3 \quad (8)$$

23.5.1 If  $E$  is equal to or greater than 0.3587  $D$ , the % of  $\text{PbSO}_4$  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  $\text{PbSO}_4$  equals 3.788  $E$ , and a new excess of  $\text{PbO}$  is calculated as follows:

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

23.6 Calculate the percent of  $\text{CO}_2$  from the analysis of Section 15 as follows:

$$G = \text{CO}_2, \% = \text{grams of CO}_2 \times 40 \quad (10)$$

23.6.1 If  $G$  is equal to or greater than 0.1314  $F$ , the percent of basic lead carbonate ( $2 \text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$ ) 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  $2 \text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$  is 8.813  $G$  and the excess  $\text{PbO}$  is:

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

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  $\text{CO}_2$  equivalent as follows: