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Designation: D126 – 87 (Reapproved 2019)

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 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:

	Sections
CHROME YELLOW, CHROME ORANGE, AND MOLYBDATE ORANGE	
Organic Colors and Lakes	7
Moisture and Other Volatile Matter	8
Matter Soluble in Water	9
Lead Chromate	10 and 11
Total Lead	12
Sulfate	13 and 14
Carbon Dioxide	15
Molybdenum	16 and 17
Extenders	18 – 22
Calculation of Substances Other than Insoluble Lead Compounds	23 and 24
PURE CHROME GREEN AND REDUCED CHROME GREEN	
Organic Colors and Lakes	25
Moisture and Other Volatile Matter	26
Matter Soluble in Water	27
Iron Blue	28
Lead Chromate	29 and 30
Barium Sulfate and Insoluble Siliceous Material	31
Total Lead	32
Sulfate	33
Calcium Oxide Soluble in Acid	34 and 35
Extenders	36
Calculation of Insoluble Lead Compounds	37
CHROMIUM OXIDE GREEN	
Organic Colors and Lakes	38
Moisture and Other Volatile Matter	39
Matter Soluble in Water	40
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:*[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.

[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.

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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 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.

[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.

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