Designation: D3256 − 86 (Reapproved 2014)

Standard Test Methods for Chemical Analysis of Phthalocyanine Blue and Green Pigments[1]

This standard is issued under the fixed designation D3256; 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.

1. Scope

1.1 These test methods cover procedures for the qualitative chemical analysis of pigments known commercially as copper phthalocyanine blue and green.

1.2 The procedures appear in the following order:

	Section
Identification	5
Moisture and Other Volatile Matter	6
Detection of Basic Dye Derivatives	7
Detection of Other Organic Coloring Matter	8
Detection of Ultramarine Blue	9
Detection of Iron Blue or Chrome Green	10

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 concerns, 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

D1193 Specification for Reagent Water

3. Significance and Use

3.1 These test methods are suitable for determining if impurities are present and establishing that the required pigments are present. These test methods may be used for manufacturing and purchasing quality control.

4. Reagents

4.1 *Purity of Reagents*—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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent grade water conforming to Specification D1193.

5. Identification

5.1 To about 0.05 g of the sample in a 50-mL beaker, add 30 mL of sulfuric acid (H_2SO_4, sp gr 1.84). Stir occasionally for 15 min, heating if necessary to accelerate the solution of the pigment. Formation of a dark greenish yellow solution indicates phthalocyanine blue and a dark reddish color indicates brominated green pigment. Pour the solution into 250 mL of water and stir. The phthalocyanine pigment should immediately precipitate as a flocculent mass.

5.2 Filter off the precipitate, washing once or twice with water. Scrape a small amount of the precipitate off the filter, place on a clean platinum wire moistened with hydrochloric acid (HCl), and subject it to the low flame of a bunsen burner. As the precipitate burns, a light blue-green flame should be clearly evident. This indicates organically combined copper.

NOTE 1—Characteristic spectrophotometric absorption spectra in the near infrared range (700 to 900 nm) are exhibited by dilute solutions of copper phthalocyanine pigments (2 to 50 mg/L) in H_2SO_4 (sp gr 1.84). The absorption maxima, being sharp and well-defined, may be used for positive qualitative identification of phthalocyanine pigments. Phthalocyanine green has maxima at approximately 820 nm and 860 nm for bromine-free types; for the chlorinated-brominated types, the maxima appear at approximately 830 to 840 nm and 875 to 885 nm.

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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.

6. Moisture and Other Volatile Matter

6.1 Determine moisture in accordance with Method A of Test Methods D280.

7. Detection of Basic Dye Derivatives

7.1 Add to 1 g of the sample, 50 mL of a mixture of equal parts of ammonium hydroxide NH_4OH (sp gr 0.90) and denatured ethyl alcohol (95 %). Warm gently and filter. Neutralize the filtrate with tartaric acid solution (200 g/L) until slightly acid to litmus. If the solution is colorless, discounting a slight yellow tinge, no basic dye is present.

7.2 If the solution is colored beyond a slight yellow tinge, add about 5 mL of 0.1 *N* titanium trichloride ($TiCl_3$) solution (Note 2). If a basic dye is present, the color will lighten significantly. If no basic dye is present, no significant color change will occur.

NOTE 2—Titanium trichloride is marketed as a 16 % solution in HCl. Mix 7 mL of this solution with 90 mL of HCl (1 + 12) to obtain a reagent approximately 0.1 *N*. Protect from oxidation.

8. Detection of Other Organic Coloring Matter

8.1 *Procedure*—Weigh about 0.05 g of the sample into each of two 50-mL beakers. Add 25 mL (at room temperature) of denatured ethyl alcohol (95 %) to one beaker and about 25 mL of acetone to the other. Stir each thoroughly for a few minutes and let stand for about 2 h. Filter through two thicknesses of medium-texture, qualitative filter paper. The presence of more than an extremely slight pink, yellow, or blue tint in either filtrate indicates other organic coloring matter is in the pigment. Organic colors resistant to the above reagents are not likely to be in phthalocyanine pigments but, if desired, procedures given in standard references can be followed to establish if they are present.

9. Detection of Ultramarine Blue

9.1 *Procedure*—Warm gently about 1 g of the sample with about 5 mL of HCl (1 + 1). Decomposition of the ultramarine blue takes place with evolution of H_2S. This may be detected by either its odor or a brown coloration appearing on a strip of moistened lead acetate paper.

10. Detection of Iron Blue and Chrome Green

10.1 *Procedure:*

10.1.1 To 1 g of the sample in a 100-mL beaker, add 25 mL of NaOH solution (50 g/L). Boil for about 1 min. Dilute to approximately 40 mL and filter.

10.1.2 Add HCl (1 + 1) to the filtrate until faintly acid to litmus. Add 2 mL of a ferric iron solution [ferric sulfate or ferric alum (20 g/L)]. The development of a blue color reveals the presence of ferrocyanide, and hence iron blue or chrome green in the original pigment. For amounts around 0.05 % iron blue, the color may take 2 to 3 h to develop.

11. Precision and Bias

11.1 No precision or bias information are available for these tests.

12. Keywords

12.1 chrome green; detection of; iron blue; detection of; phthalocyanine blue; analysis of; phthalocyanine green; analysis of; pigment; ultramarine blue; detection of