



Designation: D4450 – 85 (Reapproved 2007)

## Standard Test Method for Analysis of Zinc Hydroxy Phosphite Pigment<sup>1</sup>

This standard is issued under the fixed designation D4450; 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.

### 1. Scope

1.1 This test method is intended for the determination of zinc oxide and phosphorous acid content of the pigment commercially known as zinc hydroxy phosphite. The zinc content is determined by ethylenediaminetetraacetate (EDTA) titration and calculated as zinc oxide (ZnO), while the phosphorus content is determined colorimetrically and calculated as phosphorous acid ( $H_3PO_3$ ).

1.2 The analytical procedures appear in the following order:

	Sections
Zinc oxide	6 through 8
Phosphorous acid	9 through 14

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*:<sup>2</sup>  
[D1193 Specification for Reagent Water](#)

### 3. Summary of Test Method

3.1 The zinc is determined by dissolving the test sample in nitric acid, adjusting the pH of the solution to 5 to 5.5 and titrating with EDTA.

3.2 The phosphorus is determined colorimetrically with the aid of nitric acid, ammonium vanadate, and ammonium mo-

lybdate. The absorbance of the test sample is compared to a calibration curve that yields the number of milligrams of phosphorus.

### 4. Significance and Use

4.1 This test method may be used to confirm the stated zinc oxide and phosphorous acid content of zinc hydroxy phosphite pigment.

### 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.<sup>3</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 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification [D1193](#).

### TOTAL ZINC AS ZINC OXIDE

### 6. Reagents

6.1 *Ammonium Hydroxide* (sp gr 0.90), concentrated ammonium hydroxide ( $NH_4OH$ ).

6.2 *Glacial Acetic Acid* (min 99.7 %), concentrated glacial acetic acid ( $CH_3COOH$ ).

6.3 *Acid Ammonium Acetate Buffer*—Mix 400 mL of distilled water and 400 mL of reagent grade ammonium hydroxide (sp gr 0.90). Add 375 mL of reagent grade glacial acetic acid slowly while stirring.

6.4 *Cupric Sulfate Solution* (0.1 M)—Dissolve 25 g of  $CuSO_4 \cdot 5H_2O$  in distilled water and dilute to 1 L.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is 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> *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.