



Designation: D 4450 – 85 (Reapproved 2002)

Standard Test Method for Analysis of Zinc Hydroxy Phosphite Pigment¹

This standard is issued under the fixed designation D 4450; 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 approval. A superscript epsilon (ϵ) 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 *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:*
D 1193 Specification for Reagent Water²

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 molybdate. 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.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.

5.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

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.

6.5 *Disodium Ethylenediaminetetraacetate Dihydrate* (0.05 M) (EDTA Solution)—Dissolve 18.6 g of the reagent grade salt in distilled water and dilute to 1 L. Standardize the solution as follows:

6.5.1 Transfer 25 mL of lead standard (6.6) to a 400-mL beaker. Add concentrated ammonium hydroxide (6.1) dropwise until a permanent precipitate just forms. Add 25 mL of acid ammonium acetate (6.3), dilute to 200 mL, heat to boiling, add 4 drops of copper EDTA (6.7) and 12 drops of PAN (6.8), and titrate with EDTA to a clear yellow.

6.5.2 Calculate the molarity of the EDTA, *M*, as follows:

¹ 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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² *Annual Book of ASTM Standards*, Vol 11.01.

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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.