



Designation: D3280 – 85 (Reapproved 2014)

Standard Test Methods for Analysis of White Zinc Pigments¹

This standard is issued under the fixed designation D3280; 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 analysis of white zinc pigments.

1.2 The analytical procedures appear in the following order:

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Zinc Oxide	18
Zinc Sulfide	19
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Moisture and Other Volatile Matter	21
Barium Sulfate	22
Titanium Dioxide	23

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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

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.

D280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments
D1193 Specification for Reagent Water
D1394 Test Methods for Chemical Analysis of White Titanium Pigments
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Summary of Test Method

3.1 Zinc Oxide:

3.1.1 *Total Zinc*—Determined using diphenylamine as an internal indicator and also using uranyl acetate as an external indicator. Total impurities are calculated.

3.1.2 *Total Sulfur*—Determined as BaSO₄ and calculated to sulfur.

3.1.3 *Moisture and Volatile Matter*—Determined in accordance with Method A of Test Methods D280.

3.2 Leaded Zinc Oxide:

3.2.1 *Total Lead*—Determined as PbSO₄ and calculated to percent PbO.

3.2.2 *Total Zinc*—Determined on the filtrate from procedure in 13.2.1 in accordance with methods in Sections 7 or 8.

3.2.3 *Total Sulfur*—Determined as BaSO₄ and calculated to percent SO₃.

3.2.4 *Total Impurities*—Calculated from compositional data.

3.2.5 *Moisture and Other Volatile Matter*—Determined in accordance with Method A of Test Methods D280.

3.2.6 *Water Soluble Salts*—Determined gravimetrically.

3.3 Zinc Sulfide:

3.3.1 *Total Zinc*—Determined using uranyl acetate external indicator in accordance with Section 9.

3.3.2 *Zinc Sulfide*—Determined in accordance with Sections 8 or 9 and calculating ZnO to ZnS.

3.3.3 *Water Soluble Salts*—Determined in accordance with Section 18.

3.3.4 *Moisture*—Determined in accordance with Method A of Test Methods D280.

3.3.5 *Barium Sulfate*—The sample is treated with N₂SO₄ and Na₂CO₃ and the residue of BaCO₃ is dissolved in NCl and (NH₄)₂SO₄ added to precipitate BaSO₄, which is weighed.

3.3.6 *Titanium Dioxide*—Determined in accordance with Test Methods D1394.

4. Significance and Use

4.1 White zinc pigments find considerable use in white paints, and as such it is useful to formulators and users to be able to monitor the amounts of these pigments in whole paints. It is also of interest to raw material suppliers and paint producers to check the specifications of each pigment.

5. Reagents

5.1 *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.

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

5.3 Concentration of Reagents:

5.3.1 *Concentrated Acids and Ammonium Hydroxide*—When acids and ammonium hydroxide are specified by name or chemical formula only it should be understood that concentrated reagents of the following specific gravities or concentrations are intended:

Acetic acid, HC ₂ H ₃ O ₂	99.5 %
Hydrochloric acid, HCl	sp gr 1.19
Hydrofluoric acid, HF	48 %
Nitric acid, HNO ₃	sp gr 1.42
Sulfuric acid, H ₂ SO ₄	sp gr 1.84
Ammonium hydroxide, NH ₄ OH	sp gr 0.90

The desired specific gravities or concentrations of all other concentrated acids are stated whenever they are specified.

5.3.2 *Diluted Acids and Ammonium Hydroxide*—Concentrations of diluted acids and ammonium hydroxide, except when standardized, are specified as a ratio stating the number of volumes of the concentrated reagents to be diluted with a given number of volumes of water, as in the following example: HCl (1+99) means 1 volume of concentrated HCl (sp gr 1.19) diluted with 99 volumes of water.

6. Preparation of Sample

6.1 Grind dry pigments, if lumpy or not finely ground, to a fine powder for analysis. Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. Mix the sample in all cases thoroughly before taking specimens for analysis.

6.2 Separate pigments from paints or pastes, grind to a fine powder, pass through a 180- μ m (No. 80) sieve (**Note 1**) to remove any skins, thoroughly mix, and oven dry at 105°C. Moisten such pigments after weighing with a little alcohol before adding reagents for analysis.

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.

NOTE 1—Detailed requirements for this sieve are given in Specification **E11**.

6.3 Preserve all samples in stoppered bottles or containers.

ZINC OXIDE

7. Total Zinc, Using Diphenylamine as Internal Indicator

7.1 Reagents:

7.1.1 *Diphenylamine Indicator Solution (10 g/L)*—Dissolve 1 g of diphenylamine in 100 mL of H₂SO₄.

7.1.2 *Potassium Ferrocyanide (1 mL = 0.008 g Zn)*—Dissolve 35 g of K₄Fe(CN)₆·3H₂O in water and dilute to 1 L and add 0.3 g of potassium ferricyanide (K₃Fe(CN)₆). Standardize the solution by titrating against zinc (320 to 340 mg), following the procedure described in **7.2**. Calculate the grams of zinc equivalent to 1.00 mL of the solution.

7.2 *Procedure*—Weigh to 0.1 mg about 0.4 g of the sample into a tall form 400-mL beaker. Moisten with about 20 mL of water, and dissolve in 15 mL of HCl. Neutralize with NH₄OH, using litmus as the indicator. Add an excess of 15 mL of H₂SO₄ (1+2) and dilute to 200 mL. Heat to approximately 60°C, add 2 drops of diphenylamine indicator solution and while stirring vigorously, titrate with K₄Fe(CN)₆ solution to the color change from purple to a persistent yellowish green.

NOTE 2—The true end point is a sharp, persistent change from a purple to a yellowish green. At the beginning of the titration, a deep blue color is developed after addition of a few millilitres of K₄Fe(CN)₆ solution. About 0.5 to 1.0 mL before the true end point is reached, the solution changes from a blue to a purple color. After the purple color is developed, the titration should be continued dropwise to the persistent yellowish green end point.

7.3 *Calculation*—Calculate the percent total zinc as ZnO, *A*, as follows:

$$A = \left[\frac{V_2 Z \times 1.245}{S_1} \right] \times 100 \quad (1)$$

where:

*V*₂ = K₄Fe(CN)₆ solution required for titration of the specimen, mL,
Z = zinc equivalent of the K₄Fe(CN)₆ solution, g/mL,
*S*₁ = specimen weight, and
 1.245 = molecular weight ZnO (81.38)/molecular weight Zn (65.38).

8. Total Zinc, Using Uranyl Acetate as External Indicator

8.1 Reagents:

8.1.1 *Uranyl Acetate Indicator Solution (50 g/L)*—Dissolve 5 g of UO₂(C₂H₃O₂)₂·2H₂O in 100 mL of water and make slightly acid with acetic acid.

8.1.2 *Potassium Ferrocyanide, Standard Solution (1 mL = 0.008 g Zn)*—Prepare and standardize as in **7.1.2**. Run a blank titration with the same amounts of reagents and water. Calculate the zinc equivalent of the solution as follows:

$$Z = W/(V - B) \quad (2)$$

where:

Z = zinc equivalent of the K₄Fe(CN)₆ solution, g/mL,
W = zinc used,