Designation: D6280 – 98 (Reapproved 2014)

# Standard Specification for Zinc Phosphate Pigments[1]

This standard is issued under the fixed designation D6280; 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 This specification covers three types of pigments commercially known as zinc phosphate each of which may or may not be available in specific grades delineated by particle size or oil absorption.

1.1.1 Type I—Zinc Phosphate, dihydrate predominant.

1.1.2 Type II—Zinc Phosphate, dihydrate tetrahydrate mixture.

1.1.3 Type III—Zinc Phosphate, tetrahydrate predominant.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D153 Test Methods for Specific Gravity of Pigments

D185 Test Methods for Coarse Particles in Pigments

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

D281 Test Method for Oil Absorption of Pigments by Spatula Rub-out

D1193 Specification for Reagent Water

D1208 Test Methods for Common Properties of Certain Pigments

D1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems by Hegman-Type Gage

D2448 Test Method for Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment

## 3. Significance and Use

3.1 Zinc phosphate functions as both a chemical and a pigment. As a pigment it is used in a variety of applications including that of corrosion inhibiting paints.

## 4. Composition and Properties

4.1 Zinc phosphate pigment is a white corrosion inhibiting pigment consisting either predominately of zinc phosphate dihydrate ($Zn_3(PO_4)_2 \cdot 2H_2O$) or a mixture of zinc phosphate dihydrate and zinc phosphate tetrahydrate ($Zn_3(PO_4)_2 \cdot 4H_2O$) or predominately of zinc phosphate tetrahydrate which is free from extenders, diluents, and other pigments.

4.2 Zinc phosphate shall be a chemically prepared pigment and shall be of such type and grade as to conform to the requirements prescribed in Table 1. They shall additionally be free of extenders, modifiers, diluents, alteration of stoichiometric chemical structure, co-reacted precipitates, and carbonaceous material.

4.3 The desired properties of the pigment, other than as herein indicated, shall be subject to mutual agreement between interested parties and shall be based upon a satisfactory match between any submitted sample and a previously agreed upon reference sample.

## 5. Classification

5.1 *Type I*—which consists predominately of zinc phosphate dihydrate ($Zn_3(PO_4)_2 \cdot 2H_2O$) and exhibits a differentiating loss on ignition of the dried pigment at 600°C between 8.5 and 10.0 weight %.

5.2 *Type II*—which consists essentially of a mixture of zinc phosphate dihydrate ($Zn_3(PO_4)_2 \cdot 2H_2O$) and Zinc Phosphate Tetrahydrate ($Zn_3(PO_4)_2 \cdot 4H_2O$) and exhibits a differentiating loss on ignition of the dried pigment at 600°C between 10.0 and 14.0 weight %.

5.3 *Type III*—which consists predominately of zinc phosphate tetrahydrate ($Zn_3(PO_4)_2 \cdot 4H_2O$) and exhibits a differentiating loss on ignition of the dried pigment at 600°C between 14.0 and 18.0 weight %.

## 6. Sampling

6.1 Two samples shall be taken at random from different packages from each lot, batch, days pack or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in ratio of two samples for each 5000 kg, except for those shipments of less than 5000 kg where two samples shall be taken. At the option of the interested party the

[1] This specification 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.

Current edition approved Dec. 1, 2014. Published December 2014. Originally approved in 1998. Last previous edition approved in 2008 as D6280 – 98 (2008). DOI: 10.1520/D6280-98R14.

[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.

**TABLE 1 Zinc Phosphate Pigment Properties**

| Property | TYPE I | | | TYPE II | | | TYPE III | | |
|---|---|---|---|---|---|---|---|---|---|
| ZnO, weight percent (on ignited sample) | 62.6 - 65.1 | | | 62.6 - 65.1 | | | 62.6 - 65.1 | | |
| $P_2O_5$, weight percent (on ignited sample) | 34.9 - 37.4 | | | 34.9 - 37.4 | | | 34.9 - 37.4 | | |
| Loss on ignition, weight percent (of dried pigment) | 8.5 - 10.0 | | | 10.0 - 14.0 | | | 14.0 - 18.0 | | |
| | GRADE | | | GRADE | | | GRADE | | |
| Mean Particle Size (microns) | F | M | C | F | M | C | F | M | C |
| | < 2.5 | 2.5-5.0 | > 5.0 | same | | | same | | |
| Oil adsorption | > 30 | 30 - 15 | < 15 | same | | | same | | |
| Matter soluble in water, specific resistance, (min. ohm - cm) | 6500 | | | 6500 | | | 6500 | | |
| Moisture and other volatile matter (105 - 110°C) | 0.5 | | | 0.5 | | | 0.5 | | |
| Specific gravity, g/cm$^3$ | 3.0 - 3.5 | | | 3.0 - 3.5 | | | 3.0 - 3.5 | | |
| Hegman grind | 6 min | | | 6 min | | | 6 min | | |
| Coarse particle percent residue 325 M (45 µm) | 0.5 max | | | 0.5 max | | | 0.5 max | | |
| pH, aqueous suspension | 6 - 8 | | | 6 - 8 | | | 6 - 8 | | |

samples may be tested separately or as a composite sample formed by blending in equal quantities the samples from the same unit of production.

## 7. Test Methods

7.1 Tests shall be conducted in accordance with the following test methods. Test procedures not incorporated here and not covered by ASTM test methods shall be mutually agreed upon between the interested parties.

7.1.1 Specific Gravity—Test Methods D153, Method B.

7.1.2 Oil Absorption—Test Method D281.

7.1.3 Hegman Grind—Test Method D1210.

7.1.4 Coarse Particles—Test Methods D185.

7.1.5 pH—Test Methods D1208.

7.1.6 Specific Resistance—Test Method D2448.

7.1.7 Moisture—Test Methods D280.

7.1.8 Chemical Analysis—Incorporated in this specification as Annex A1 and Annex A2.

7.1.9 Loss on Ignition—Incorporated in this specification as Annex A3.

## 8. Keywords

8.1 analytical; zinc; zinc phosphate

# ANNEXES

**(Mandatory Information)**

## A1. TEST METHOD FOR DETERMINATION OF ZINC CONTENT FOR ZINC PHOSPHATE TYPE PIGMENTS

### A1.1 Scope

A1.1.1 This test method covers the determination of the zinc content for zinc phosphate monohydrate, dihydrate, tetrahydrate, or mixtures of these various crystal water content pigments.

A1.1.2 *This standard does not purport to address all the safety concerns, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish the appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### A1.2 Reference Documents

A1.2.1 *ASTM Standards:* Test Method for Determination of Loss on Ignition for Zinc Phosphate Type Pigments

D1193 Specification for Reagent Water

### A1.3 Summary of Test Method

A1.3.1 A weighed sample taken from Annex A3, Test Method for Determination of Loss on Ignition for Zinc Phosphate Type Pigments completed analysis (that is, sample contains no associated crystal water), is dissolved in ammonia buffer, complexed with disodium ethylendiaminetetraacetate dihydrate (EDTA) and back titrated with a standard zinc solution to an eriochrome black *T* endpoint where the percent ZnO is determined.

### A1.4 Significance and Use

A1.4.1 This test method provides a reliable means for determination of the percent ZnO for zinc phosphate pigments. The percent ZnO content for the product gives evidence of the chemical purity of the pigment.

### A1.5 Reagents and Materials

A1.5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in this test, 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. 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.

A1.5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II of Specification D1193.

A1.5.3 *Buffer Solution (pH 10)*—350 ml of concentrated $NH_4OH$ + $NH_4Cl$ and $H_2O$ to give 1000 mL.

A1.5.4 *Eriochrome Black T Indicator (0.5 %)*—0.25 g eriochrome Black T + 2.2 g hydroxylamine hydrochloride per 50 mL methanol.

A1.5.5 *Primary Standard Zinc Oxide (0.2 N solution)*—Accurately weigh 4.0690 g of oven dried ZnO. Dissolve in 250 mL of the buffer solution and dilute to 500 mL. (Conversely, you can weigh up approximately 4.0 g of ZnO, create the solution, and standardize it using the primary standard grade ZnO.)

A1.5.6 0.2 N *Disodium Ethylendiaminetetraacetate Dihydrate (EDTA)*—37.2 g of EDTA per litre of aqueous solution.

### A1.6 Procedure

A1.6.1 Weigh approximately 0.25 g (see Annex A3) in duplicate to the nearest 0.1 mg. Place in respective Erlenmeyer flasks. Add 25 mL of the pH 10 buffer solution. Stir gently to dissolve. Pipet 50.00 mL of the EDTA solution into each respective Erlenmeyer flask. Dilute to 200 mL with dionized water. Add 6 to 7 drops of eriochrome Black T indicator. Titrate with the 0.2 *N* ZnO solution to a wine - red endpoint. Run a blank by titrating 50.00 mL EDTA containing 25 mL of pH 10 buffer solution with the 0.2 *N* ZnO solution.

### A1.7 Calculations

A1.7.1 Calculate the percent ZnO as follows:

$$\% \text{ ZnO} = \frac{(V_b - V_s) \times N \text{ ZnO} \times 4.069}{\text{Mass of Sample}} \qquad \text{(A1.1)}$$

where:

$V_b$ = ZnO for blank, mL,
$V_s$ = ZnO for sample, mL,
$N$ = normality of the zinc solution, and
4.069 = factor for conversion to % ZnO.

A1.7.2 Report the mean, estimated standard deviation, and coefficient of variation for the analysis.

### A1.8 Precision and Bias

A1.8.1 *Precision*—Results should be considered suspect if the standard deviation is greater than 0.2 %.

A1.8.2 In an repeatability study of this test method, in which two samples containing 50.5 to 52.0 % ZnO were analyzed by one operator per test, the observed standard deviation for 100 separate tests was determined to be 0.066.

A1.8.3 *Bias*—Bias does not apply because there is no material of acceptance value available.

## A2. TEST METHOD FOR DETERMINATION OF PHOSPHATE CONTENT FOR ZINC PHOSPHATE TYPE PIGMENTS

### A2.1 Scope

A2.1.1 This test method covers the determination of the phosphate content for zinc phosphate monohydrate, dihydrate, tetrahydrate, or mixtures of these various crystal water content pigments.

A2.1.2 *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.*

### A2.2 Reference Documents

A2.2.1 *ASTM Standards:* Test Method for Determination of Loss on Ignition for Zinc Phosphate Type Pigments

D1193 Specification for Reagent Water

### A2.3 Summary of Test Method

A2.3.1 A weighed sample taken from Annex A3, Test Method for Determination of Loss on Ignition for Zinc Phosphate Type Pigments completed analysis (that is, sample contains no associated crystal water), is dissolved in ammonia,

and the liberated phosphate precipitated with ammonium molybdate. The resulting precipitate is filtered through a Gooch or sintered glass crucible and dried to constant weight where the percent $P_2O_5$ is determined.

### A2.4 Significance and Use

A2.4.1 This test method provides a reliable means for determination of the percent $P_2O_5$ for zinc phosphate pigments. The percent $P_2O_5$ content for the product gives evidence of the chemical purity of the pigment.

### A2.5 Apparatus

A2.5.1 *Analytical Balance,* capable of weighing to the nearest 0.1 mg.

A2.5.2 *Oven,* capable of maintaining a temperature of 105 °C for 2 h or longer.

A2.5.3 *Gooch* or medium porosity sintered glass crucibles.

A2.5.4 *Spatula Tongs.*

A2.5.5 *Desiccator,* with drying agent.

### A2.6 Reagents and Materials

A2.6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in this test, 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. 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.

A2.6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II of Specification D1193.

A2.6.3 *Concentrated Ammonium Hydroxide,* $NH_4OH$.

A2.6.4 *Concentrated Nitric Acid,* $HNO_3$.

A2.6.5 *Ammonium Nitrate,* $NH_4NO_3$.

A2.6.6 *Ammonium Molybdate* (Johnson's Formula) : Mix 55 g of $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ and 50 g of $NH_4NO_3$ with 18 mL of $NH_4OH$ and 20 mL of $H_2O$. Stir. Dilute to 700 mL with $H_2O$, and heat with occasional stirring until all salts have dissolved. Dilute to 1000 mL. Let stand overnight. Filter the solution using a fine grade filter paper but do not wash the residue.

### A2.7 Procedure

A2.7.1 Weigh approximately 2.0 g (see Annex A3) of the test pigment in duplicate to the nearest 0.1 mg. Place in a 250-mL glass stoppered Erlenmeyer flask. Add 25 mL of $NH_4OH$, stopper, and swirl over a period of 60 min. Add 25 mL of $H_2O$ and filter through fine filter paper into a 400-mL beaker, washing the residue well with water. Neutralize the filtrate with $HNO_3$ (requires approximately 35 mL). Add 15 mL of $HNO_3$ and 6 g of $NH_4NO_3$. Stir. Heat the clear solution to 80°C (do not exceed) and add 75 mL of the ammonium molybdate solution with constant stirring. Stir for several minutes and let the precipitate settle for a minimum of 2 h. Filter the precipitate through a tared gooch or medium glass crucible. Wash the precipitate well with 1 % $HNO_3$, then give a final wash with water. Dry the crucible and its contents for 2 h at 105°C in an oven. Cool the crucible in a desiccator and determine the weight of the precipitate to the nearest 0.1 mg.

### A2.8 Calculations

A2.8.1 Calculate the percent $P_2O_5$ as follows:

$$\%\ P_2O_5 = \frac{\text{mass PPT} \times 3.783}{\text{mass sample}} \qquad \text{(A2.1)}$$

A2.8.2 Report the mean, estimated standard deviation, and coefficient of variation for the analysis.

### A2.9 Precision and Bias

A2.9.1 *Precision*—Results should be considered suspect if the standard deviation is greater than 0.2 %.

A2.9.2 *Bias*—See previous statement given in A1.8.3.

## A3. TEST METHOD FOR DETERMINATION OF LOSS OF IGNITION OF ZINC PHOSPHATE TYPE PIGMENTS

### A3.1 Scope

A3.1.1 This test method covers the determination of the loss on ignition in zinc phosphate monohydrate, dihydrate, tetrahydrate, or mixtures of these various crystal water content pigments.

### A3.2 Summary of Test Method

A3.2.1 A sample of zinc phosphate is dried at 110°C for 1 h to constant weight.

A3.2.2 A weighed portion of the previously dried zinc phosphate with associated crystal water is ignited in a muffle oven at 600°C for 30 min and the crystal water amount is determined by difference.

### A3.3 Significance and Use

A3.3.1 This test method provides a reliable means for determining the associated crystal water content on zinc phosphate pigments, and its respective classification type.

### A3.4 Apparatus

A3.4.1 *Analytical Balance,* capable of weighing to the nearest 0.1 mg.

A3.4.2 *Muffle Furnace,* capable of maintaining a temperature of 600°C for 30 min or longer.

A3.4.3 *Porcelain Crucibles.*

A3.4.4 *Spatula Tongs.*

A3.4.5 *Desiccator,* with drying agent.

### A3.5 Procedure

A3.5.1 Dry a 5-g sample at 110°C for 1 h or to constant weight.

A3.5.2 Weigh to the nearest 0.1 mg approximately 2 to 3 g of the previously dried pigment sample into a previously ignited and weighed crucible. Heat the crucible in the muffle oven at 600°C for 30 min. Cool the crucible in a desiccator and weigh. Repeat the heating, cooling and weighing sequence until the change in mass between two successive weighings does not exceed 1.0 mg. Perform the analysis in duplicate.

### A3.6 Calculations

A3.6.1 Calculate the percent loss on ignition as follows:

$$\% \text{ Loss on ignition} = \frac{M_0 - M_1}{M_0} \quad \text{(A3.1)}$$

where:

$M_0$ = mass of the test pigment before ignition and
$M_1$ = mass of the test pigment after ignition.

A3.6.2 Report the mean, estimated standard deviation, and coefficient of variation.

### A3.7 Precision and Bias

A3.7.1 *Precision*—Results should be considered suspect if the estimated standard deviation is greater than 0.1 %.

A3.7.2 *Bias*—See previous statement given in A1.8.3.