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Standard Specification for Zinc Phosphate Pigments¹

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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.

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

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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.

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 ($\text{Zn}_3(\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$) or a mixture of zinc phosphate dihydrate and zinc phosphate tetrahydrate ($\text{Zn}_3(\text{PO}_4)_2 \cdot 4\text{H}_2\text{O}$) 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 ($\text{Zn}_3(\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$) 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 ($\text{Zn}_3(\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$) and Zinc Phosphate Tetrahydrate ($\text{Zn}_3(\text{PO}_4)_2 \cdot 4\text{H}_2\text{O}$) 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 ($\text{Zn}_3(\text{PO}_4)_2 \cdot 4\text{H}_2\text{O}$) and exhibits a differentiating loss on ignition of the dried pigment at 600°C between 14.0 and 18.0 weight %.

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 ₂ O 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		
Mean Particle Size (microns)	GRADE			GRADE			GRADE		
	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		

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 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, health, and environmental 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 ethylenediaminetetraacetate 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,³ 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 $\text{NH}_4\text{OH} + \text{NH}_4\text{Cl}$ 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 Ethylenediaminetetraacetate 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 ionized 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:

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

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.

³ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.