



Designation: D 3969 – 01

Standard Test Method for Zirconium in Paint Driers by EDTA Method¹

This standard is issued under the fixed designation D 3969; 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 test method covers the titrimetric determination of zirconium in zirconium driers used in the coatings industry and utilizes the disodium salt of ethylenediaminetetraacetic acid dihydrate (EDTA).

1.2 This test method is limited to the determination of the zirconium content of a liquid zirconium drier that does not contain other drier elements. The test method is not applicable to drier blends and does not differentiate hafnium from zirconium.

1.3 All cations that can be titrated with EDTA in acid media interfere and must not be present in the sample.

1.4 This test method has been tested for concentrations of 6 and 12 % zirconium, but there is no reason to believe that it is not suitable for higher or lower zirconium concentrations, provided specimen size is adjusted proportionately.

1.5 *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 600 Specification for Liquid Paint Driers²

D 1193 Specification for Reagent Water³

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals⁴

E 300 Practice for Sampling Industrial and Specialty Chemicals⁵

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 D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Jan. 10, 2001. Published April 2001. Originally published as D 3969 – 80. Last previous edition D 3969 – 85 (1994).

² *Annual Book of ASTM Standards*, Vol 06.04.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

⁵ *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.

3. Summary of Test Method

3.1 The zirconium drier is digested with concentrated sulfuric acid and 30 % hydrogen peroxide to destroy all organic matter. The diluted solution is boiled with an excess of EDTA, the pH adjusted, and the excess titrated with bismuth nitrate using xylenol orange as the indicator.

4. Significance and Use

4.1 The amount of zirconium drier used in oxidizing-type coatings significantly affects their drying properties. This test method may be used to confirm the stated content of a pure liquid zirconium drier manufactured for use by the coatings industry.

5. Apparatus

5.1 *Centrifuge*, capable of developing 1000 to 2000 g.

6. Reagents

6.1 *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.⁵ 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

6.3 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated, ammonium hydroxide (NH₄OH).

6.4 *Bismuth Nitrate, Standard Solution (0.05 M)*—Dissolve 24.25 g of bismuth nitrate (Bi(NO₃)₃·5H₂O) in 20 to 30 mL of concentrated nitric acid (use a magnetic stirrer if possible). After solution is complete, dilute to 1 L with water.

NOTE 1—Add the water very slowly to the acid solution, while cooling the flask in an ice bath to prevent splattering.

6.5 *EDTA, Standard Solution (0.05 M)*—Dissolve 18.61 g of EDTA in 300 to 500 mL of water and dilute to 1 L. Store in a polyethylene or borosilicate glass bottle.