



Designation: D3804 – 02 (Reapproved 2021)

## Standard Test Method for Iron in Paint Driers by EDTA Method<sup>1</sup>

This standard is issued under the fixed designation D3804; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the titrimetric determination of iron in liquid iron driers soluble in isopropyl alcohol and utilizes the disodium salt of ethylenediaminetetraacetic acid dihydrate (EDTA).

1.2 This test method is limited to the determination of the iron content of a liquid drier that does not contain other drier elements. This method is not applicable to drier blends.

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

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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:*<sup>2</sup>

**D600 Specification for Liquid Paint Driers** (Withdrawn 2021)<sup>3</sup>

**D1193 Specification for Reagent Water**

**E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals** (Withdrawn 2009)<sup>3</sup>

**E300 Practice for Sampling Industrial Chemicals**

<sup>1</sup> 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.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

### 3. Summary of Test Method

3.1 The liquid iron drier is diluted with isopropyl alcohol and the iron chelated with excess standard EDTA. The solution is buffered and the excess EDTA is titrated with standard zinc chloride solution to the Eriochrome Black T end point.

### 4. Significance and Use

4.1 This test method may be used to confirm the stated content of a liquid iron drier soluble in isopropyl alcohol and manufactured for use in the coatings industry. The content determines activity level.

### 5. Interferences

5.1 All cations that can be titrated with EDTA in alkaline media interfere and must not be present in the sample or must be masked.

### 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.<sup>4</sup> 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 **D1193**.

6.3 *Ammonium Hydroxide (1+3)*—Add 10 mL of concentrated ammonium hydroxide (NH<sub>4</sub>OH, sp gr 0.90) to 30 mL water.

6.4 *Buffer Solution*—Add 350 mL of concentrated ammonium hydroxide (NH<sub>4</sub>OH) to 54 g of ammonium chloride (NH<sub>4</sub>Cl) and dilute to 1 L with water.

<sup>4</sup> *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.

6.5 *EDTA, Standard Solution (0.01 M)*—Weigh to 10 mg about 3.73 g of the disodium salt of ethylenediaminetetraacetic acid dihydrate (EDTA), dissolve in water, and dilute to approximately 1 L in a polyethylene or borosilicate glass bottle.

6.6 *Hydrochloric Acid (1+3)*—Add 3 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) to 9 mL of water.

6.7 *Eriochrome Black-T Indicator*—Titrate 0.20 g of the concentrated dye with 100 g of NaCl and store in a tightly stoppered jar. This mixture remains stable for several years.

6.8 *Isopropyl Alcohol (99.5 %)*.

6.9 *Zinc Chloride, Standard Solution (0.01 M)*—Weigh to 0.5 mg about 0.65 g of zinc (**Note 1**) onto a glazed paper. Transfer to a 1-L volumetric flask and add 25 mL of dilute HCl (7+18) (add 7 mL of concentrated acid (sp gr 1.19) to 18 mL of water). Warm if necessary on a steam bath to dissolve completely. Cool, dilute to the mark with water and mix thoroughly. Calculate the exact molarity of this approximately 0.01-*M* solution as follows:

$$M_2 = W/65.4 \quad (1)$$

where:

$M_2$  = molarity of ZnCl<sub>2</sub> solution, and  
 $W$  = zinc used, g.  
 65.4 = atomic weight of zinc.

**NOTE 1**—Zinc ribbon cut into small pieces with clean scissors is preferred. Granular (20 mesh) zinc requires several hours of heating on a steam bath for complete solution. Store the zinc ribbon in a tightly sealed container to prevent the surface of the zinc from oxidizing.

## 7. Sampling

7.1 Take a small sample of liquid drier from bulk using the procedures in Practice **E300** appropriate for the size of container, tanks and tank cars or drums and cans.

**NOTE 2**—Liquid driers are normally homogeneous so that only simple physical tests, such as specific gravity or solids content, on top and bottom samples from tanks are required to confirm that separation has not occurred. Agitate drums in accordance with Practice **E300**.

7.2 Examine the sample of drier for sediment or suspended matter which if present is evidence of noncompliance with Specification **D600**.

7.3 If the sample is homogeneous keep it in a stoppered vessel to prevent solvent evaporation prior to analysis.

## 8. Standardization

8.1 *EDTA, Standard Solution (0.01 M)*—Transfer 40.0 mL of this solution from a buret into a 250-mL assay beaker or wide-mouthed flask. Add 50 mL of isopropyl alcohol, 10 mL of buffer solution, and 0.2 g of indicator (6.7). Mix thoroughly by swirling. Titrate with standard ZnCl<sub>2</sub> solution (6.9) to the first permanent tinge of red. Calculate the exact molarity of this approximately 0.01 *M* solution as follows:

$$M_1 = V_2 M_2 / V_1 \quad (2)$$

where:

$M_1$  = molarity of EDTA solution,  
 $V_2$  = ZnCl<sub>2</sub> solution, mL,

$M_2$  = molarity of ZnCl<sub>2</sub> solution, and  
 $V_1$  = EDTA solution, mL.

## 9. Procedure

9.1 Check the clarity of the drier. If not clear, centrifuge a portion of the sample until it is clear, keeping the centrifuge tube stoppered to prevent solvent evaporation.

9.2 Place a few grams of the drier in a 50-mL Erlenmeyer flask fitted with a cork through which passes a dropping tube and rubber bulb (or medicine dropper) and obtain the total weight. Weigh by difference to 0.5 mg, 0.18 to 0.22-g specimens (8 to 12 drops), into 400-mL beakers. This specimen size is for driers of 6 % iron content; adjust the size according to expected percent iron to contain about 0.2 mM of iron. Add 100 mL of isopropyl alcohol and 3 mL 1 + 3 HCl to each specimen and swirl to mix. Add a few boiling aids and heat the solution just to boiling on a hot plate; remove and cool to room temperature in a water bath.

9.3 From a buret measure 40.0 mL of standard EDTA solution into each beaker. Neutralize with dilute NH<sub>4</sub>OH (1 + 3), as indicated by a change in the color of the solution from yellow to reddish. Add 10 mL of the buffer solution and 0.3 g of the Eriochrome Black-T indicator mixture. This addition should result in a blue-colored solution. Immediately back-titrate the excess EDTA with the standard ZnCl<sub>2</sub> solution (**Note 3**) to the first permanent tinge of red (**Note 4**). The back-titration must be completed within 2 min (**Note 5** and **Note 6**).

**NOTE 3**—During the titration stir the solution manually or by means of a magnetic stirrer.

**NOTE 4**—To some observers, this color change appears as a change to purple. However, the transition is sharp and, with a little practice, easily noted.

**NOTE 5**—The time used in the titration step with ZnCl<sub>2</sub> solution affects the results. A titration time of less than 2 min gives consistently good results. Longer times give higher results.

**NOTE 6**—If the end point is overstepped, add 1.0 mL of the EDTA solution to the mixture and titrate again with standard ZnCl<sub>2</sub> solution. Use total volume of each solution for the calculation.

## 10. Calculation

10.1 Calculate the percent of iron present as follows:

$$\text{Iron, \%} = (V_3 M_1 - V_4 M_2) 5.59 / S \quad (3)$$

where:

$V_3$  = EDTA solution, mL,  
 $M_1$  = molarity of EDTA solution,  
 $V_4$  = ZnCl<sub>2</sub> solution, required for specimen, mL,  
 $M_2$  = molarity of ZnCl<sub>2</sub> solution,  
 $S$  = sample used, g, and  
 5.59 = millimolar weight of Fe × 100

## 11. Precision and Bias<sup>5</sup>

11.1 The precision estimates are based on an interlaboratory study in which one operator in seven different laboratories

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1021. Contact ASTM Customer Service at service@astm.org.