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# Standard Test Method for Lead in Paint Driers by EDTA Method[1]

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## 1. Scope

1.1 This test method covers a titrimetric determination of lead in liquid paint driers that can be dissolved in glacial acetic acid and utilizes the disodium salt of ethylenediaminetetraacetic acid dihydrate (EDTA).

1.2 This test method is not applicable to drier blends. If driers other than cobalt are present, they may interfere by reacting with EDTA under the conditions used for analysis.

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

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

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:*[2]

D600 Specification for Liquid Paint Driers

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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

E300 Practice for Sampling Industrial Chemicals

## 3. Summary of Test Method

3.1 The liquid drier is dissolved in glacial acetic acid, diluted with isopropyl alcohol and water, and treated with an excess of standard EDTA solution. The excess is titrated with standard cupric sulfate solution using PAN as the metal indicator.

## 4. Significance and Use

4.1 The amount of lead drier used in oxidizing-type coatings significantly affects their drying properties. This test method may be used to confirm the stated lead content of pure liquid lead drier soluble in glacial acetic acid and 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.[4] 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 Chloride* ($NH_4Cl$).

6.4 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide ($NH_4OH$).

6.5 *Buffer Solution* (pH 10.0)—Dissolve 67.5 g of $NH_4Cl$ in water, add 570 mL of concentrated $NH_4OH$ (sp gr 0.90), and dilute to 1 L.

6.6 *Cupric Sulfate, Standard Solution* (0.05 *M*)—Dissolve 12.5 g of cupric sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) in water and dilute to 1 L.

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[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.

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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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] *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.

6.7 *Eriochrome Black-T Indicator*—Triturate 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 *EDTA, Standard Solution* (0.05 *M*)—Dissolve 18.62 g of EDTA in water and dilute to 1 L. Store in a polyethylene or borosilicate glass bottle.

6.9 *Glacial Acetic Acid* (sp gr 1.06).

6.10 *Isopropyl Alcohol* (99.5 %).

6.11 *Methyl Red Indicator*—Dissolve 0.2 g of methyl red in 100 mL of methanol, ethanol, or isopropanol.

6.12 *PAN Indicator (1(2-Pyridylazo)-2-Naphthol)*—Prepare a dilute solution of the indicator by dissolving 0.10 g in 100 mL of ethanol or isopropanol.

6.13 *Sodium Acetate Solution*—Prepare a saturated aqueous solution of sodium acetate ($NaC_2H_3O_2$) in water.

6.14 *Sodium Chloride* (NaCl).

6.15 *Sodium Hydroxide Solution (80 g/L)*—Dissolve 80 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

6.16 *Sulfuric Acid (5+1)*—Carefully mix 8 volumes of concentrated sulfuric acid ($H_2SO_4$, sp gr 1.84) with 1 volume of water.

6.17 *Zinc Oxide* (ZnO).

## 7. Sampling

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

NOTE 1—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 the section on Tube Sampling of 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 Weigh to the nearest 0.1 mg approximately 1.5 g of ZnO into a 100-mL beaker and add 10 mL of $H_2SO_4$ (8 + 1). Transfer the solution to a 500-mL volumetric flask and dilute to the mark with water. Pipet a 50-mL aliquot into a 500-mL Erlenmeyer flask, add 100 mL of distilled water, 2 or 3 drops of methyl red solution, and neutralize with NaOH solution (80 g/L). Add 5 mL of buffer solution, 0.2 g of Eriochrome Black-T indicator, and titrate with EDTA until the color changes from red to blue, where the last red shade just disappears.

8.1.1 Calculate the molarity, $M_1$, of the EDTA solution as follows:

$$M_1 = \frac{W}{(V_1 \times 0.814)} \quad (1)$$

where:

$W$ = ZnO used, g,
$V_1$ = EDTA solution, mL, and
$0.0814$ = molecular weight of ZnO × $10^{-3}$
= millimolar weight of ZnO.

8.2 *$CuSO_4$, Standard Solution* (0.05 *M*)—Determine the titer of the $CuSO_4$ solution by titrating 50 mL of the standard EDTA solution with $CuSO_4$ as described in 9.4 using the PAN indicator.

8.2.1 Calculate the molarity, $M_2$, of the $CuSO_4$ solution as follows:

$$M_2 = \frac{(M_1 \times V_2)}{V_3} \quad (2)$$

where:

$M_1$ = molarity of EDTA solution,
$V_2$ = EDTA solution, mL, and
$V_3$ = $CuSO_4$ 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. Keep the centrifuge tube stoppered so that solvent will not evaporate.

9.2 Weigh the following specimen amounts into a 500-mL Erlenmeyer flask and add 5 mL of glacial acetic acid: (*1*) for 24 % lead, 1.0 to 1.5 g, (*2*) for 30 and 32 % lead, 0.7 to 0.9 g and (*3*) for more or less lead use proportionate specimen sizes. Warm on a hot plate at low heat, swirling the flask until the specimen is completely dissolved.

9.3 In a second flask (blank) put the same amount of glacial acetic acid but no specimen. Add 200 mL of isopropyl alcohol, 100 mL of water, 6 mL of concentrated $NH_4OH$, 10 mL of $Na_2C_2H_3O_2$, and 50 mL of EDTA solution to each flask. Swirl to ensure complete mixing and warm both flasks at steam bath temperature for 5 min.

9.4 To each of the flasks add 2 mL of PAN indicator and titrate with the standard $CuSO_4$ solution to a blue end point.

NOTE 2—Because of the color of metal indicators (and some of their complexes) being affected by pH changes, the pH must be kept constant during titration by the recommended buffer during titration.

## 10. Calculation

10.1 Calculate the percent of lead, *A*, present as follows:

$$A = \left[ \frac{(B - V_4) \times M_2 \times F}{S} \right] \times 100 \quad (3)$$

where:

$B$ = $CuSO_4$ solution required for titration of the blank, mL,
$V_4$ = $CuSO_4$ solution required for the specimen, mL,
$M_2$ = molarity of $CuSO_4$ solution,
$F$ = millimolecular weight of the metal in the sample
= 0.2072 for lead, and
$S$ = specimen used, g.

## 11. Precision and Bias (see Practice E180)

11.1 *Precision:*

11.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 0.10 % lead.

11.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories, should be considered suspect if they differ by more than 0.50 % lead.

11.2 *Bias*—Bias cannot be determined for this method because there are no accepted standards for lead in paint driers.

## 12. Keywords

12.1 drier analysis; EDTA method; lead

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