Designation: D 2613 – 85 (Reapproved 1994)$^{\epsilon 1}$

Designation: D 2613 – 01

Standard Test Method for Calcium or Zinc in Paint Driers by EDTA Method[1]

This standard is issued under the fixed designation D 2613; 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.

ε[1] NOTE—Section 11 was editorially corrected in June 1994.

[1] This test method is under the jurisdiction of ASTM Committee D-1 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 Nov. 29, 1985. Jan. 10, 2001. Published January 1986. March 2001. Originally published as D 2613 – 67 T. Last previous edition D 2613 – 785 (1994)ε1.

1. Scope

1.1 This test method covers a titrimetric determination of calcium in liquid calcium driers and zinc in liquid zinc driers that can be dissolved in a toluene-alcohol mixture and utilizes the disodium salt of ethylenediaminetetraacetic acid dihydrate (EDTA).

1.2 This test method is not applicable to drier blends.

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

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 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 Driers[2]

D 1193 Specification for Reagent Water[3]

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[4]

E 300 Practice for Sampling Industrial Chemicals[4]

3. Summary of Test Method

3.1 The liquid calcium or zinc drier is dissolved in toluene and ethyl alcohol and treated with an excess of standard EDTA solution. The excess is titrated with a standard zinc chloride solution using Eriochrome Black-T as the indicator.

4. Significance and Use

4.1 This test method may be used to confirm the stated calcium or zinc content of pure liquid calcium or zinc driers soluble in toluene-alcohol 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.[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.

[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. Pharmacopeutieal Convention, Inc. (USPC), Rockville, MD.

6.3 *Buffer Solution*—Add 350 mL of concentrated ammonium hydroxide (NH_4OH, sp gr 0.90) to 54 g of ammonium chloride (NH_4Cl) and dilute to 1 L with water.

6.4 *EDTA, Standard Solution (0.05 M)*— Weigh 18.62 g of EDTA, dissolve in water and dilute to 1 L. Store in a polyethylene or borosilicate glass bottle.

6.5 *Indicator Mixture*—Triturate 0.2 g of Eriochrome Black-T and 100 g of sodium chloride (NaCl) and store the mixture in a tightly stoppered bottle. This mixture remains stable for several years.

6.6 *Zinc Chloride, Standard Solution (0.05 M)*—Weigh 3.2690 g of zinc metal to 0.5 mg and dissolve in 50 mL of dilute hydrochloric acid (14 mL of concentrated HCl (sp gr 1.42) to 36 mL of water). Warm if necessary. Dilute to 1 L in a volumetric flask.

7. Sampling

7.1 Take a small sample of liquid drier from bulk using the procedures in Practice E 300 appropriate for the size of the container: Section 19 for tanks and tank cars or Section 23 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 E 300.

7.2 Examine the sample of drier for sediment or suspended matter which if present is evidence of noncompliance with Specification D 600.

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

8. Standardization

8.1 *Zinc Chloride Standard Solution (0.05 M)*—Calculate the exact molarity, M_1, of the solution as follows:

$$M_1 = S_1/65.37 \quad (1)$$

where:
S_1 = zinc used, g, and
65.37 = zinc to produce a 1 *M* solution, g/L.

8.2 *EDTA, Standard Solution (0.05 M)*— Measure 40.0 mL of the EDTA solution into a 250-mL flask that contains 10 mL of toluene and 100 mL of 95 % ethyl alcohol pure or denatured. Add 15 mL of buffer solution, 0.2 g of indicator mixture, and mix thoroughly. Titrate with the standard zinc solution to the first permanent appearance of a red color.

8.2.1 Calculate the molarity of the EDTA solution, M_2, as follows:

$$M_2 = (V_1 \times M_1)/40.0 \quad (2)$$

where:
V_1 = $ZnCl_2$ solution, mL,
40.0 = EDTA solution titrated, 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 the solvent will not evaporate.

9.2 From a buret place a few grams of the drier in a 50-mL Erlenmeyer flask that is fitted with a cork through which passes a dropping tube and rubber bulb or eye dropper and obtain the total weight. Weigh by difference two or three 1-g specimens (10 drops weigh about 0.2 g) to the nearest 0.5 mg into 400-mL assay beakers or wide-mouth flasks (Note 2). Add 10 mL of toluene to each specimen and swirl to mix. Add 100 mL of 95 % ethyl alcohol and swirl again until the specimen is dissolved and well dispersed. From a buret measure 40.0 mL of EDTA solution into each beaker. Add 15 mL of buffer solution and 0.20 g of the indicator mixture (more or less can be added, if desired). Mix thoroughly by swirling. Titrate with the standard $ZnCl_2$ solution to the first permanent tinge of red. Maintain vigorous swirling during the titration to ensure thorough mixing of the two phases which may appear.

NOTE 2—If a magnetic stirrer is available, it is convenient to titrate in an ordinary beaker. Stirring magnetically ensures thorough mixing during the titration.

NOTE 3—If the end point is overstepped, add 1.0 mL of the EDTA solution to the mixture and titrate again with standard $ZnCl_2$ solution. Use total volume of each solution for the calculation.

10. Calculation

10.1 Calculate the percent of Ca, *A*, or Zn, *B*, as follows:

~~$A = [4.01]/S$~~ (3)

$$A = [4.01]/S \quad (3)$$

~~$B = [\ 6.54]/S$~~ (4)

$$B = [\ 6.54]/S \quad (4)$$

where:

V_2 = EDTA solution, mL,
V_3 = $ZnCl_2$ solution required for specimen, mL,
S = specimen used, g,
4.01 = millimolar weight of Ca × 100, and
6.54 = millimolar weight of Zn × 100.

11. Precision and Bias (see Practice E 180)†

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 0.05 % calcium or zinc.

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 0.10 % calcium or zinc.

11.2 *Bias*—Bias ~~has not been~~ cannot be determined because there are no accepted standards for ~~this test method.~~ calcium and zinc in paint driers.

12. Keywords

12.1 calcium; drier analysis; EDTA analysis; ~~zinc~~ zinc

~~† Editorially corrected.~~