



## Standard Test Method for Citrate in Detergents<sup>1</sup>

This standard is issued under the fixed designation D 4608; 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.

<sup>ε1</sup> NOTE—Keywords were added editorially in February 1995.

### 1. Scope

1.1 This test method covers a potentiometric titration procedure for the determination of citrate in liquid and powder detergents.

1.2 *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.* Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage. Specific safety precautions are given in Section 8.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode<sup>2</sup>
- E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals<sup>2</sup>

### 3. Summary of Test Method

3.1 The sample is titrated in an aqueous solution buffered at pH 8.5, with standard copper sulfate solution. The endpoint is detected potentiometrically using a copper ion selective electrode. The citrate content is calculated from the amount of standard copper sulfate solution consumed.

### 4. Significance and Use

4.1 This test method is suitable in research, development, and manufacturing control to monitor the level of citrate, a sequestering agent, in powder and liquid detergents.

4.2 Accurate determination of sequestering agent is important in evaluating cost and performance of detergent products.

### 5. Interferences

5.1 Other complexing agents such as nitrilotriacetic acid (NTA), ethylenedinitrilotetraacetic acid (EDTA), phosphates,

etc. will titrate as citrate in this method. For accurate citrate determinations, such complexing agents must be absent.

### 6. Apparatus

- 6.1 *pH Meter*, with millivolt capability.<sup>3</sup>
- 6.2 *Copper Ion Selective Electrode*.<sup>4</sup>
- 6.3 *Calomel Reference Electrode*.<sup>5</sup>
- 6.4 *Glass Electrode Triple Purpose*.<sup>6</sup>
- 6.5 *Buret*, semi-micro, 25 mL capacity with 0.1 mL graduations.
- 6.6 *Magnetic Stirrer*, and TFE-fluorocarbon-coated magnetic stirring bars.

### 7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>7</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.

7.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean distilled water or water of equal purity.

7.3 *Hydrochloric Acid Solution (1 + 1)*—Slowly mix 1 volume of concentrated hydrochloric acid (sp gr 1.19) with 1 volume of water.

7.4 *Sodium Hydroxide Solution (20 %)*—Prepare a 20 % aqueous solution of sodium hydroxide.

7.5 *Sodium Hydroxide Solution (0.1 N)*—Prepare a 0.1 N aqueous solution of sodium hydroxide.

<sup>3</sup> An automatic titrator may be used.

<sup>4</sup> Orion Model 94-29A or equivalent has been found suitable for this purpose. Available from Orion Inc.

<sup>5</sup> Orion Model 90-22 or equivalent has been found suitable for this purpose. Available from Orion Inc.

<sup>6</sup> Corning Model No. 476022 or equivalent has been found suitable for this purpose. Available from Corning Inc.

<sup>7</sup> *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 Pharmacopoeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-12 on Soaps and Other Detergents and is the direct responsibility of Subcommittee D12.12 on Analysis of Soaps and Synthetic Detergents.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 15.05.