Designation: D 4954 – 89 (Reapproved 1995)ε1

Standard Test Method for Determination of Nitrilotriacetates in Detergents[1]

This standard is issued under the fixed designation D 4954; 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—Keywords were added editorially in February 1995.

1. Scope

1.1 This test method describes the determination of nitrilotriacetates (NTA) in 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.* For specific safety precautions, see 6.5.

2. Summary of Test Method

2.1 A known excess amount of copper nitrate solution is added to the detergent sample under controlled pH conditions. The uncomplexed copper is back-titrated potentiometrically with a standard NTA solution using a copper ion specific electrode. The millimoles of NTA in the sample are equivalent to the millimoles of copper added, less the millimoles of the NTA titrant.

3. Significance and Use

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

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

4. Interferences

4.1 The presence of substances that will make copper complex like citrate, formate, ethylenediaminetetraacetate, and phosphonate, will interfere positively in the determination of NTA.

5. Apparatus

5.1 *Volumetric Flasks*, 200-mL, 500-mL, 1000-mL.

5.2 *Beakers*, 200-mL (tall form), 250-mL, 1000-mL.

5.3 *Pipets*, 25-mL, 100-mL.

5.4 *Graduated Cylinders*, 10-mL, 25-mL.

5.5 *Copper Ion Electrode (Orion Model 94-29)*.

5.6 *Single Junction Reference Electrode (Orion Model 90-01)*, filled with Orion 90-00-01 solution.

5.7 *pH Combination Electrode*.

5.8 *Burets*, 10-mL, 25-mL, 50-mL.

5.9 *pH Meter*, with millivolt capabilities.

5.10 *Automatic Titrator*—may be substituted for 5.8 and 5.9.

5.11 *Magnetic Stirrer/Hot Plate*.

5.12 *Balance*, with 1-mg sensitivity.

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 specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[2] Other grades may be substituted, 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 *Disodium Ethylenediaminetetraacetate (EDTA)*, 0.1 M.[3]

6.3 *Nitric Acid*, concentrated.

6.4 *Sodium Hydroxide*, 50 % reagent solution.

6.5 *Sodium Hydroxide*, 20 % solution. Mix 40 g of the 50 % solution with 60 g of water. Cool. Wear a face shield during mixing.

6.6 *Cupric Nitrate*, 0.1 M. Weigh (to the nearest 0.1 g) 6.3 g of copper metal and transfer into a 600-mL beaker. Add about 100 mL of water. Place a magnetic bar into the beaker and place on a stirrer/hotplate. Add 30 mL of concentrated nitric acid while stirring and heat gently. Stir until the copper metal dissolves. Add more nitric acid if undissolved metal remains. After dissolution, cool and transfer quantitatively to a 1-L volumetric flask. Dilute to the mark and mix the solution well.

6.6.1 Alternatively, weigh out (to the nearest 1 g) 23 g of reagent grade cupric nitrate, 2½ hydrate crystals, and transfer to a 600-mL beaker. Add 400 mL of water. Stir to dissolve the crystals and add 5 mL of concentrated nitric acid. Transfer to a 1-L volumetric flask, dilute to the mark and mix well.

6.7 *Trisodium Nitrilotriacetate Monohydrate (NTA)*, 0.3 M. Weigh out (to the nearest 1 g) 41 g of NTA and dissolve in 400

[1] 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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[2] American Chemical Society, 1155 16th St., NW, Washington, DC 20036.

[3] EDTA solution is available from J. T. Baker Inc., already standardized against NIST Reference Material Calcium Carbonate.