



Designation: D4954 – 89 (Reapproved 2025)

Standard Test Method for Determination of Nitrilotriacetates in Detergents¹

This standard is issued under the fixed designation D4954; 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.

1. Scope

1.1 This test method describes the determination of nitrilotriacetates (NTA) in detergents.

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

1.3 *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.* For specific safety precautions, see 6.5.

1.4 *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. 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.

¹ This test method is under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergents and is the direct responsibility of Subcommittee D12.12 on Analysis and Specifications of Soaps, Synthetics, Detergents and their Components.

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² *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.