



Designation: **D4008 – 95 (Reapproved 2009) D4008 – 16**

## Standard Test Method Guide for Measuring Anti-Soil Deposition Properties of Laundry Detergents (Not Suitable for Detergent Ranking)<sup>1</sup>

This standard is issued under the fixed designation D4008; 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.

### 1. Scope

1.1 This test method provides guidelines for measuring the ability of detergents to prevent the deposition of soils from detergent solutions onto fabrics. It is intended as a laboratory screening test to aid in the formulation of detergent products, for quality control, and as a basis between the purchaser and seller in standardizing specific products' performance.

1.2 The anti-soil deposition performance of detergent products will vary greatly depending on the type of soils and fabrics used in the test. Therefore, selection of the soils, fabrics, reference detergents, and test conditions shall be made by agreement between the interested parties on the basis of experience.

1.3 The values stated in either inch-pound or SI units are to be regarded separately as the standard. The values given in parentheses are for information only.

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.* Material safety data sheets are available for reagents and materials. Review them for hazards prior to usage.

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

~~D1193 Specification for Reagent Water~~

~~D2960 Guide for Controlled Laundering Test Using Naturally Soiled Fabrics and Household Appliances (Withdrawn 2013)~~<sup>3</sup>

~~E97 Method of Test for Directional Reflectance Factor, 45-Deg 0-Deg, of Opaque Specimens by Broad-Band Filter Reflectometry (Withdrawn 1991)~~<sup>3</sup>

~~E313 Practice for Calculating Yellowness and Whiteness Indices from Instrumentally Measured Color Coordinates~~

### 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *reflectance retention*—the change in reflectance between clean fabric and the same fabric after laundering.

3.1.2 *soil deposition*—the soiling of clean fabrics by soil that has been added to the wash bath rather than by soil removed from another fabric.

3.1.3 *soil redeposition*—the soiling of clean, or relatively clean, fabrics during the laundering process by soil that has been removed from another fabric.

### 4. Summary of Test Method

4.1 Swatches of clean fabrics are washed, a minimum of three times, in solutions of the test detergent prepared to contain known amounts of particulate and oily soils.

4.1.1 Identical clean swatches are similarly exposed to prepared solutions of a suitable reference detergent containing an identical soil load. The ability of the test detergent to prevent soil deposition is estimated by comparing the reflectance retention of the swatches washed in the test detergent to the reflectance retention of those washed in the reference detergent.

<sup>1</sup> This test method guide is under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergents and is the direct responsibility of Subcommittee D12.15 on Physical Testing.

Current edition approved Oct. 1, 2009 July 1, 2016. Published December 2009 August 2016. Originally approved in 1981. Last previous edition approved in 2003 2009 as D4008 – 95(2003);(2009). DOI: 10.1520/D4008-95R09:10.1520/D4008-16.

<sup>2</sup> 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.

<sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

## 5. Significance and Use

5.1 The test as now constituted is not suitable for ranking of detergent products, since no basis is available at this time for correlation of the anti-deposition performance of detergents using any particular combination of soils and fabrics with anti-redeposition performance during washing of naturally soiled articles.

5.2 A suggested procedure for comparing the performance of any two laundry detergents or naturally soiled family items in home laundry equipment, under controlled conditions on a paired comparison basis, is described in Method **D2960**.

## 6. Apparatus

6.1 *Laboratory Washer*—A laboratory-scale, agitator-type washing machine.<sup>4</sup>

6.2 *Reflectometer*, calibrated by means of standard vitreous enamel plaques having reflectances in the range of the fabric or swatch samples being measured.

## 7. Reagents and Materials

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 the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>5</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, references to water shall be understood to mean Type IV reagent water conforming to Specification **D1193**.

7.3 *Standard Soils*—Soils suitable for deposition-type detergency tests are not commercially available, therefore, laboratory prepared soils must be used. For example, vacuum cleaner dirt, air conditioner filter dirt, and colored clay material, or mixtures of two or more of these can be used as particulate soil. Triglyceride, fatty acid, paraffinic hydrocarbon, and fatty alcohol are examples of oily soils that may be used. Mixtures of two or more of these oily soils or a mixture based on the components of human sebum may also be used. The oily soils may be added as emulsions or solvent solutions or they may be added after first mixing with the particulate soil in a solvent solution and subsequent evaporation of the solvent.

NOTE 1—A 1:1 mixture of vacuum cleaner dirt and Bandy Black clay has been found suitable as a particulate soil. A suggested use level of this soil is 0.5 g/1000 mL of wash solutions. The vacuum cleaner dirt can be obtained from domestic or office locations. Before use, it is screened through a 150-mesh (Tyler equivalent) screen to remove lint, trash, and coarse particles.

A 1:1 mixture of triolein (technical) and mineral oil (Saybolt viscosity 340 to 350) has also been found satisfactory. A suggested use level is 0.100 g/1000 mL of wash solution.

An artificial sebum composition has also been found suitable as an oily soil. A useful formula for such a composition was reported by Spangler, W. G., Cross, H. D., and Schaafsma, B. R., *Journal of the American Oil Chemists' Society*, Vol 42, 1965, p. 723. A suggested use level is 0.100 g/1000 mL of wash solution.

7.3.1 The following procedure is a satisfactory method for convenient addition of particulate and oily soils to the wash bath: Thoroughly blend together 20 g of Bandy Black clay and 20 g of vacuum cleaner dirt. Add this mixture to 150 mL of methylene chloride containing 4 g of triolein and 4 g of mineral oil. While constantly stirring this mixture, evaporate the solvent under a heat lamp in a hood. The oily/particulate soil is gently ground in a mortar and pestle to disperse any lumps, that may have formed. Store the soil mixture at refrigerator temperature until use. Recommended use level of this combined soil is 0.600 g/1000 mL of wash solution.

7.4 *Standard Clean Fabrics*<sup>6</sup>—Because different fabrics have different soiling characteristics depending on their surface chemistry and morphology, it is recommended that several fabric types be used. For example, cotton, nylon, polyester, and polyester/cotton blends may be used. These should be cut to 4 by 4½ in. in size with pinked, merrowed, or fused edges to prevent unraveling. Ten swatches of each fabric are used for each test. Test each fabric type separately. Pay special attention to the identification of finishes, for example durable-press, soil-release, etc.

NOTE 2—A preliminary laundering of the fabric may be desired to remove any material that might interfere with the test.

7.5 *Reference Detergent*—A comparison detergent suitable to the fabric and conditions shall be included in each run.

7.6 *Hard-Water Stock Solution*—Prepare a hard-water stock solution by dissolving  $2.940 \pm 0.002$  g of calcium chloride dihydrate ( $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ ) and  $2.033 \pm 0.002$  g of magnesium chloride hexahydrate ( $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ) in about 300 mL of water. Dilute to a volume of 1 L with additional water. This solution contains 3000 ppm hardness (expressed as calcium carbonate) with a Ca + Mg molar ratio of 2:1. Other ratios may be used.

7.7 *Calcium/Magnesium Ratio (as  $\text{CaCO}_3$ )*—It is suggested that this ratio be adjusted for different water hardnesses as follows:

<sup>4</sup> The Terg-o-tometer has been found satisfactory.

<sup>5</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 Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

Water Hardness Range, ppm (ppg)	Calcium/ Magnesium Ratio
0 to 60 (0 to 3.5)	4:1
61 to 120 (3.6 to 7.0)	3:1
121 and over (7.1 and over)	2:1

These ratios are based on calculations derived from data included in Public Water Supplies of 100 Largest Cities in the United States, by E. Becker and C. N. Durfor; U. S. Government Printing Office, 1964.

## 8. Sampling

8.1 Samples of the detergent to be tested and of the reference detergent shall be obtained from homogeneous lots and stored in nearly filled, airtight containers until tested.

## 9. Reflectance Determinations

9.1 Operate the reflectometer in accordance with the instructions supplied with the instrument for 45°, 0° luminous reflectance using a procedure that avoids fluorescence effects. Standardize the instrument with standard vitreous enamel plaques that have reflectances closest to the reflectances of the swatches being measured. Use sufficient layers of the fabric being read, so that addition of another layer of fabric does not cause a change in the reflectance of the swatch being read. Make a sufficient number of reflectance readings on the clean swatches to give the desired statistical level of significance. Repeat after the swatches have been washed and dried. Procedures used in making reflectance determinations shall conform to those of Test Method E97. Reflectance readings may be recorded in terms of *Rd*, *L*, or *Y*. (See Test Method E313, Appendix A1.)

## 10. Procedure

10.1 Operate the washer at a suitable fixed speed (90 cycles/min is suggested). Maintain the water bath at a temperature suitable to the fabric, detergent, and conditions of the test. Prepare duplicate solutions of the test product and the reference detergent in 1000 mL of water at the desired concentration, temperature, and water hardness. It is recommended that concentrations of 0.1, 0.15, 0.2, and 0.25 %, temperatures of 70, 100, and 130°F (21, 38, and 54°C), and water hardnesses of 35, 150, and 300 ppm be included. Transfer the solutions to the buckets of the washer. Add to each bucket the selected soils, start the wash agitator, and agitate the solution for 2 min to fully disperse the soil. Without stopping the agitation, add the selected fabrics and wash for 10 min. Then remove the swatches, squeeze them by hand, and transfer to a bucket containing 1000 mL of water of the same hardness at the same temperature and rinse 3 min. Then damp dry the swatches between clean toweling and air dry. Repeat the above tests at least two more times using the same swatches. Determine the reflectance of the washed and dried swatches.

NOTE 3—In any test program a water hardness of 150 ppm should be included, since approximately 80 % of the families in the U.S. have this level of hardness, or below. It should also be noted that while natural soils add hardness to the wash bath, artificial soils may not, therefore a hardness of 75 ppm may be more realistic for the lower hardness level.

NOTE 4—To avoid discoloration, change in structure of the swatch surfaces, or both, do not use direct heat or pressure from an instrument such as a photographic print dryer, hand iron, automatic ironer, or tumble dryer.

## 11. Calculation

11.1 Calculate the percent reflectance retention as follows:

$$\text{Reflectance retention, \%} = (C_w/C_o) \times 100 \quad (1)$$

where:

$C_w$  = average reflectance of the test swatches after washing, and

$C_o$  = average reflectance of the test swatches before washing.

11.2 Calculate the reflectance retention of the test detergent as the percent of the reference detergent as follows:

$$\text{percent of reference} = \frac{RR_{\text{test}}}{RR_{\text{ref}}} \times 100 \quad (2)$$

where:

$RR_{\text{test}}$  = percent reflectance retention of the test detergent, and

$RR_{\text{ref}}$  = percent reflectance retention of the reference detergent.

## 12. Precision and Bias

12.1 Because of the variability of soils and fabrics used in this test, it does not have the precision or reproducibility of an analytical determination.

12.2 Adequate reproducibility is possible if soils, fabrics, and reference detergent are not varied and reflectance retention of the test detergent is reported as percent of the reference detergent.