

# Standard Guide for Measuring Anti-Soil Deposition Properties of Laundry Detergents<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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

- 1.1 This guide provides direction 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 There is no single or combination of oily soils or particulate soils, or both, that will precisely predict overall the performance of a product or treatment with respect to anti-redeposition as perceived by a consumer. This guide is intended to assess or compare detergent performance with respect to the anti-redeposition performance for the specific soil chosen.
- 1.4 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.
- 1.5 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

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of Test for Directional Reflectance Factor, 45-Deg 0-Deg, of Opaque Specimens by Broad-Band Filter Reflectometry (Withdrawn 1991)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 Guide

- 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>&</sup>lt;sup>1</sup> This 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 July 1, 2016. Published August 2016. Originally approved in 1981. Last previous edition approved in 2009 as D4008 – 95(2009). DOI: 10.1520/D4008-16.

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

## 5. Significance and Use

5.1 The guide can be used to compare anti-redeposition performance of products; however, there is no confirmed basis for correlation of this controlled laboratory technique with consumers' ranking of anti-redeposition performance.

### 6. Apparatus

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

### 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—Laboratory prepared soils or commercially available soils may 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—A1:1 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.

A1:1 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

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

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—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 may be cut to 4 by 4½ in. in size with pinked, merrowed, or fused edges to prevent unraveling. Multiple swatches of each fabric should be 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, if desired.
- 7.6 Hard-Water Stock Solution—Prepare a hard-water stock solution by dissolving  $2.940 \pm 0.002$  g of calcium chloride dihydrate (CaCl<sub>2</sub>·2H<sub>2</sub>O) and  $2.033 \pm 0.002$  g of magnesium chloride hexahydrate (MgCl<sub>2</sub>·6H<sub>2</sub>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 CaCO<sub>3</sub>)—It is suggested that this ratio be adjusted for different water hardnesses as follows:

|                                 | Calcium/  |
|---------------------------------|-----------|
|                                 | Magnesium |
| Water Hardness Range, ppm (gpg) | 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 If using a reflectometer, operate the instrument 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

<sup>&</sup>lt;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.

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.)

9.2 If using a Spectrophotometer, operate the instrument in accordance with the instructions supplied with the instrument for D65,  $10^{\circ}$  using a procedure that avoids fluorescence effects. Standardize the instrument based on its recommended procedure. Use sufficient layers of the fabric being read, so that the 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. Color or color difference calculations from your reference fabric may be recorded in terms of  $dE^*$ ,  $L^*$ , CIE Whiteness Index or Ganz Whiteness Index.

#### 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. Be aware that results may be impacted due to temperature and water hardness differences. For a holistic view of your product performance with respect to anti-redeposition, it is recommended that temperatures of 70, 90, and 120°F (21, 32, and 49°C), and water hardnesses of 35, 120, 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 to illustrate the products build-up profile. Determine the reflectance of the washed and dried swatches.

Note 3—In any test program a water hardness of 120 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. Calculations

11.1 Calculate the percent reflectance retention as follows:

Reflectance retention, 
$$\% = (C_w/C_a) \times 100$$
 (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:

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

where:

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

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

11.3 Calculate the total color difference (dE\*) between the standard fabric and your test fabric as follows:

$$\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2} \tag{3}$$

11.4 Whiteness can be calculated by a variety of indices. Many indices are available within today's instrument's software. Below are some options:

(1) ASTM Practice E313

WI CIE = Y + (800)(.3138 - x) + (1700)(0.3310 - y)Where Y, x, y are luminance factor and the chromaticity values of the swatch.

(2) WIGanz = Y - (1868.322 x) + (-3695.690 y) + 1809.441 Where Y, X, y are luminance factor and the chromaticity values of the swatch.

#### 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.



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