

Designation: D 4265 – 98

# Standard Guide for Evaluating Stain Removal Performance in Home Laundering<sup>1</sup>

This standard is issued under the fixed designation D 4265; 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 is a guide for evaluating stain removal performance of home laundry products or home laundering conditions. It provides guidance for the selection, preparation, application, and examination of various types of stains on test fabrics that are subjected to controlled but practical stain removal treatment conditions.

1.2 There is no single type of stain that will predict the overall performance of a product or treatment method. A single test, even with a variety of stains, can only predict how products or treatment methods compare under the particular conditions chosen for evaluation. A series of assessments is necessary to evaluate the many aspects of stain removal performance and to simulate consumer experience more closely.

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 and health practices and determine the applicability of regulatory limitations prior to use.

# 2. Referenced Documents

# <u>ASTM D</u>

2.1 ASTM Standards: eh.al/catalog/standards/sist/d77f4e8

- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials<sup>2</sup>
- D 2960 Test Method of Controlled Laundering Test Using Naturally Soiled Fabrics and Household Appliances<sup>3</sup>

## 3. Terminology

3.1 Definitions:

3.1.1 *home laundering*—the cleaning and restoring of textile materials to a serviceable condition, using the washing and drying equipment commonly found in the home.

3.1.2 *stain*—a local area of undesired foreign matter on a textile material, differing in appearance from the textile substrate.

<sup>2</sup> Annual Book of ASTM Standards, Vol 06.01.

## 4. Summary of Guide

4.1 Stains are artificially applied in a standardized manner to specified fabric substrates. After a prescribed time for setting the stains, the test swatches are treated with the products or procedures being compared or both, and the relative degree of removal is assessed.

## 5. Significance and Use

5.1 This guide suggests a number of staining agents that are representative of stains commonly encountered in household laundry. The assessment need not be limited to this suggested list of stains, especially if special product types or end uses are being evaluated. However, even when the assessment incorporates a wide variety of stains, an overall performance ranking of products or treatment methods cannot be presumed since there is no confirmed basis for correlation of this controlled laboratory technique with consumers' ranking of stain removal performance.

5.2 The evaluations generated by this guide should be regarded as diagnostic screening tests that are useful in formulation studies, quality control, and ingredient raw material qualification. This guide provides considerable flexibility in choosing specific stains, washing conditions, and laboratory equipment appropriate to the objective of the evaluation. This procedural latitude may result in a reduced level of interlaboratory precision and such comparison of results must be evaluated with caution.

5.3 The procedure is applicable to all types of home laundry products including detergents, presoak and prespotter products, bleaches, and detergent boosters. It is not intended for the evaluation of products or conditions normally associated with commercial laundering or dry cleaning establishments.

## 6. Selection and Preparation of Fabric Swatches

6.1 The relative effectiveness of various laundry products or procedures for removing different types of stains will be influenced by the nature of the fabric substrate. Testing should encompass more than one type of fabric, with results tabulated separately for each fabric type. The fabric types most common in U. S. laundry are polyester/cotton blends with permanent press finish, all cotton, all polyester, and nylon.

NOTE 1-When ordering pre-cut swatches, the vendor must be alerted that identification of the face side and grain (thread direction) of the

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<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 15.04.

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swatches is an important testing criterion. These factors must also be considered when bulk yardage is purchased from any source by the experimenter for eventual cutting into test specimen swatches.

6.2 All fabric test swatches should be washed prior to application of stains. This is done to eliminate residues of mill finishing agents which might influence stain removal results and also to better simulate the fabric condition when staining incidents occur in household use.

6.2.1 Sufficient yardage of test fabric is pre-washed to coverthe needs of the test series. The washing is more efficiently done before the cloth is cut into individual swatches, but can be accomplished with the pre-cut swatches if this is the fabric form that has been purchased.

6.2.2 Minimum pre-washing conditions would be two wash cycles in a household automatic washer (see Note 2) with 140  $\pm$  5 °F (less than 3 grains) water and ambient (less than 3 grains) than water rinses. The first cycle should include a recommended usage level of detergent (AATCC Standard Detergent or the detergent being used in the test) and 1 cup (240 mL) liquid hypochlorite bleach. The second wash cycle would include 60 g of a complex phosphate water conditioner and no detergent or bleach.

NOTE 2—Any brand of household automatic washer is acceptable as a standard washing machine if it has the following operating characteristics on the normal setting: water fill, 16 to 19 gal; agitator speed,  $70 \pm 5$  opm; spin speed, 500 to 510 rpm; washing time, 12 to 15 min.

6.2.3 Dry the test cloth 35 to 40 min at  $150 \pm 10^{\circ}$ F in a home dryer after the last wash cycle (see Note 3).

NOTE 3—Any brand of household automatic electric dryer is acceptable as a standard electric tumble dryer if it has the following operating characteristics: controlled exhaust temperature, ranging between 50 and 71 °C (140 and 160 °F); and a cooling period of at least 5 min while tumbling at the end of the drying circle.

6.2.4 Wash carrier towels or ballast fabrics to be used with the stained test swatches in accordance with 6.2.2, but separately from the test fabrics.

6.3 Iron the fabric on permanent press setting if necessary to obtain a flat, smooth surface.

6.4 Cut washed test fabric into desired size swatches.

6.4.1 Orientation of the test swatch can influence the observed staining/wicking characteristics of the test stain and also the stain removal perceived by either visual grading or instrumental reflectance. It is therefore important to identify the fabric grain (direction of the threads relative to the selvage edge) and the face side of the fabric when the test specimens are made. Devise a swatch marking system to assure that all specimens in the same test series are stained and graded with the same fabric orientation.

6.4.2 Size of the test swatch is not critical, though swatches of about 15 to 30 in.<sup>2</sup>(100 to 200 cm<sup>2</sup>) in area are suitable for all phases of the testing procedure. To maintain orientation consistency, use rectangular rather than square swatches.

#### 7. Stain Materials

7.1 The spectrum of selected staining materials can greatly influence the relative performance assessment of the stain removal products. The stain types most appropriate for a specific test series are usually dictated by experience. Staining materials most often recommended for stain removal assessments are tea, coffee, wine, grape juice, mustard, spaghetti sauce, beef gravy, blueberry puree or jam, chocolate syrup, grass, human or beef blood, ball–point pen ink, facial makeup base, dirty motor oil, artificial sebum/particulate soil, and clay. See Annex A1, Annex A2, and Annex A3 for more detailed descriptions and preparation information for these staining materials.

7.2 A minimum of at least six stain types is suggested for characterizing the performance profile of a stain removal product, especially if more than one functional ingredient (for example, oxidizing agent, enzyme, solvent, etc.) is present in the tested formulations.

#### 8. Stain Application on Swatches

8.1 General Techniques for Spotting:

8.1.1 The intent is to have the fabric swatch suspended horizontally in air with the center of the swatch relatively taut when the staining material is applied. Any mounting device that will achieve these criteria is acceptable. Suggested devices include wooden or cardboard pinning frames, glass beakers (>2 in. diameter) with the swatch secured over the top by rubber bands, and glass bottles with the swatch secured within the top opening by a plastic retaining ring.

8.1.2 The size of the circular stain pattern should be a minimum of 2 in. in diameter to accommodate instrument grading. Many staining agents will wick beyond the original application area. A4 by 5 in. test swatch is suggested if wicking to the edges of the fabric is not desired. Wide variations in wicking patterns across test swatches can increase judgement variability when the treated swatches are graded visually.

8.1.3 Using a capillary pipet, medicine dropper, metal spatula or 2-in die cut polyurethane sponge (depending on viscosity of the staining material), place an appropriate amount of staining material in the center of the suspended test swatch. The amount of stain material that will adequately cover the intended spot area will vary by fabric substrate and with different batches of stain material. Some experimentation will be needed on the first few swatches; these trial swatches should then be discarded. See Annex A3 for further tips on stain application techniques.

8.1.4 Keep each freshly stained swatch suspended in its pinning frame or place it face–side–up on a disposable plastic cup until stain is dry (usually 2 to 3 h). Do not allow stained specimens to contact each other in a manner that would transfer stains.

#### 8.2 Aging of Stains:

8.2.1 When the aqueous-based stains have thoroughly air dried (feel dry to the touch), set individual swatches in a dark place at room temperature ( $70 \pm 5$  °F) for a minimum of 1 day. The aging time for oil-based stains may need to be shortened due to excessive wicking on some substrates. A minimum of 4 h aging is recommended for oily stains. If wicking to the swatch edge still occurs in this shortened period, use a larger swatch for oily stains or reduce the amount applied.

8.2.2 Wrap swatches intentionally held for more than one day's aging in aluminum foil and store in 35–40 °F controlled temperature. Swatches with dry aqueous–based stains can be

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stacked in bundles for storage, but store oily stains separately to avoid cross-wicking between test specimens.

8.2.3 Even when stored as prescribed in 8.2.2, many stains will continue to set and become increasingly less responsive to the test removal treatments. The experimenter may deliberately choose to age the stains more than 1 day before conducting the test washings. It is important, however, that all stain specimens in a test series have the same aging history and that this aging time be one of the recorded fixed variables of the test design. Experience has shown that stained swatches should be discarded if more than several weeks old.

8.2.4 Visually inspect stained swatches selected for a particular testing series for comparable appearance. Any that show atypical application or wicking patterns should not be selected.

8.2.5 Place identifying code markings on the face side of each swatch (same side as stain application) to ensure comparable orientation of the swatches when they are graded. Use a solvent–and–wash–resistant marking ink for coding.

#### 9. Procedure

9.1 Design the test protocol to generate at least three replicated values for each type of test specimen subjected to the individual variable (product or procedure) under study. For example, in one test run of a washing condition using six different stains on three fabric types, there would be a minimum of 54 stained swatches (6 stains  $\times$  3 types of fabric  $\times$  3 replications) plus nine unstained reference swatches (3 types of fabric  $\times$  3 replicates). This allows an adequate estimate of within–run variability of the results. A second and preferably, a third run must be done, generating six (or nine) values for each type of stained test specimen. The number of replications (swatches per run or number of runs) can be expanded according to the needs of the experimenter to raise the confidence level in the analysis of results between treatments.

9.2 The mixing of different kinds of stains in the same wash treatment is acceptable. However, the mixture of stain types and total number of stained swatches (soil load) should be the same in each treatment wash.

9.2.1 Other test protocol factors may present a constraint on the number of test swatches per wash load (for example, a maximum in the amount of pre-treatment product entering the wash bath). Accommodating test designs should still have the stain type and soil level variables balanced across the treatment washes.

9.3 The number of unstained swatches to be included in each test treatment for reflectance blanks should be appropriate for the test design (see 11.2).

9.4 *Fixed Test Conditions*—All test conditions not under study should be fixed within the range of normal household practice. If only one test is being run, the following conditions are suggested as representative of many households:

9.4.1 *Matched Washing Machines*—All washings must be performed in the same machine or in mechanically matched units of the same model machine.

9.4.2 *Washer Settings*—Regular cycle; appropriate water fill for clothes load size; 12 min wash period; regular spin speed. 9.4.3 *Water Characteristics; Wash and Rinse*:

9.4.3.1 *Temperature*—90  $\pm$  2 °F wash cycle; ambient water rinses.

9.4.3.2 *Hardness*—35 ppm (2 grains/gal); 100 ppm (6 grains/gal); 150 ppm (9 grains/gal) (see Note 4); and 260 ppm (15 grains/gal).

NOTE 4-Suggested hardness if only one level is tested.

9.4.3.3 The calcium/magnesium ratio of the hardness minerals (expressed as  $CaCO_3$ ) should be adjusted for different water hardnesses as follows:

Vater Hardness Range	Ca/Mg Ratio
0 to 60 ppm	4:1
61 to 120 ppm	3:1
121 and over	2:1

9.4.4 *Fabric Load*—Minimum of 4 lb. Supplement test swatches with ballast fabrics, such as pillow cases, terry towels, or commercially available yard goods cut in pieces no larger than  $1 \text{ yd}^2$  per piece. When running successive test loads, use fresh ballast or rewash ballast for each load. Natural or artificially soiled ballast will add realism to the testing conditions, but a high level of testing variability will be introduced unless this factor is rigorously standardized.

9.4.5 *Product Concentrations*—Use products according to manufacturer's label recommendations. Reduce recommended level of detergent added to the wash by the amount used in pretreating (see 9.4.6).

9.4.6 Pretreating Methods (if applicable)—Equalize contact time for all stains, with a min of 1 min before fabric is introduced to the agitating wash water. If prespotter label directions suggest rubbing, the testing should be done both with and without a standardized rubbing step. If not specifically recommended by product manufacturer, suggested test amounts for prespotting are: 2 mL for sprayed–on pre– spotters (see Note 5); 5 mL for liquid detergents; and 1 tsp (enough to cover stain) for pastes of dry detergents made with sufficient water to give it a toothpaste–like consistency.

NOTE 5—Liquid prespotting products packed in aerosol containers are not sprayed onto the stain, but are applied as a liquid without propellant. Spray into suitable container to collect appropriate quantity needed for the test treatments.

9.4.7 Order of Addition to Wash Cycle:

9.4.7.1 Fill with water and start agitation,

9.4.7.2 Add washing products; and

9.4.7.3 Add test fabrics.

9.4.8 *Machine Drying Conditions*—high setting (160  $\pm$  10 °F max outlet temperature) for 45  $\pm$  5 min. Air drying, out of direct sunlight, is also acceptable.

9.4.9 Miscellaneous Guides:

9.4.9.1 Attaching test swatches to suitable carrier towels is strongly advised for operational efficiency and to simulate more normal mechanical agitation and spin drying action in the washing machine and to minimize test swatch wrinkling. Test specimens are most appropriately attached by one edge around the perimeter of the carrier towel. The carrier towel should not be positioned as a backing for the test swatch. Attachment can be accomplished by sewing, by noncorrosive pins or staples, or plastic fasteners commonly used for apparel hang tags.

9.4.9.2 Wrinkled swatches may be manually smoothed; avoid pressing the swatches with an iron.