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Standard Guide for Evaluating Cleaning Performance of Ceramic Tile Cleaners[1]

This standard is issued under the fixed designation D5343; 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—Editorial updates were made in March 2012.

1. Scope*

1.1 This guide covers the evaluation of the cleaning performance of products intended for use on ceramic tiles. This guide provides techniques for soiling, cleaning, and evaluating performance of detergent systems under controlled, but practical, hard-surface cleaning conditions.

NOTE 1—The soils described in this guide are known in the industry as "soap scum."

1.2 Such systems include any detergent, cleaner, or abrasive cleanser intended for cleaning hard surfaces composed of ceramic tile. This guide is not appropriate for evaluating performance on grout. This guide also excludes other surfaces such as glass, resilient flooring, synthetic countertop surfaces or washable wall surfaces. The products for which this guide is intended include solutions of soluble powdered detergent, dilutions of concentrated liquid detergent, or products intended to be used full strength, for example, foams, sprays, liquid, wipes, powders, or paste.

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.* Material Safety Data Sheets are available for reagents. Review them for hazards prior to usage.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1729 Practice for Visual Appraisal of Colors and Color Differences of Diffusely-Illuminated Opaque Materials

D2960 Guide for Controlled Laundering Test Using Naturally Soiled Fabrics and Household Appliances (Withdrawn 2013)[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *bathroom soil*—the soil composed of materials present on typical bathroom surfaces.

3.1.2 *cycle*—a cycle is defined as being both the back and forth strokes on the scrubbing apparatus.

3.1.3 *soil*—in hard surface cleaning, foreign matter on a hard surface such as a ceramic tile.

3.1.4 *substrate*—the soiled surface that is being cleaned.

4. Summary of Guide

4.1 Soils are artificially applied in a standardized manner to a ceramic tile surface. The soiled surfaces are cleaned using a straight-line washability apparatus, and the cleaned substrates are evaluated using reflectance measurements, or visually by a panel of judges. A schematic diagram of the soil composition is summarized in Fig. 1.

5. Significance and Use

5.1 This guide suggests methodology for cleaning tests. This methodology can only be applied to assess product performance on typical bathroom soils and is not inclusive of all potential soils present on ceramic tiles or other bathroom surfaces. An assessment of cleaning performance on surfaces other than ceramic tile cannot be presumed since there is no confirmed basis for correlation for this soil on other surface or substrate types.

5.2 The results of tests based on this guide are regarded as diagnostic screening values useful in formulation studies, quality control, and ingredient raw material qualification. The results of this guide should be compared to control treatments, which are incorporated into each performance evaluation. These results should be considered to be relative to **all** other treatments in the study and are not absolute values. For interlaboratory comparisons, exact treatment conditions must

[1] This guide is under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergents and is the direct responsibility of Subcommittee D12.16 on Hard Surface Cleaning.

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[2] 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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

***A Summary of Changes section appears at the end of this standard**

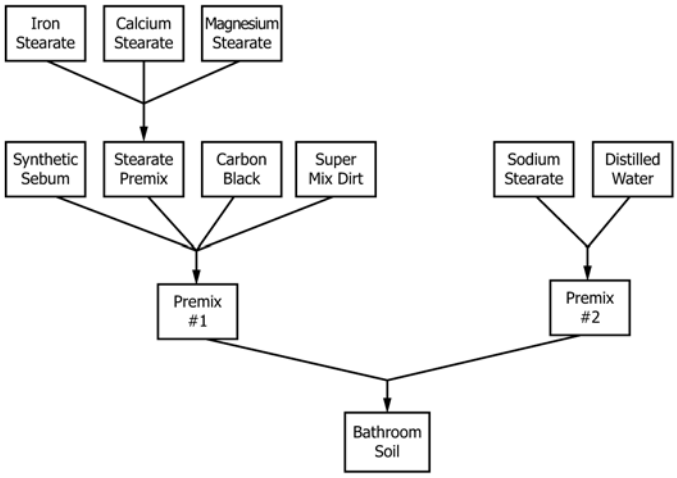

FIG. 1 Bathroom Soil Composition

be established and then results should be compared as relative ranking to the control treatments rather than as absolute values.

5.3 This guide is applicable to testing all types of multi-purpose household and bathroom cleaners, whether the detergent is prepared by dissolving a soluble powder, a dilutable liquid, or is a non-dilutable product. It may also be useful for evaluation of products or conditions normally associated with industrial or institutional cleaners.

6. Preparation of Soil

6.1 *Summary of Method*—White ceramic tiles are soiled with a composite soil composed of two separate preblends. One preblend consists of a mixture of synthetic sebum, metal stearates, carbon black, and dirt. The second preblend is a predissolved sodium stearate solution. Soil is applied using a painting pad applicator. The soiled tiles can be stored for up to three months when stored at 35°F (1.67°C). The soiled substrate is scrubbed with a fine-celled sponge, to which the detergent system being tested has been applied, or with a damp sponge when the product is to be applied to the soil surface. Cleaning performance is evaluated by comparing reflectance measurements made on the clean/unsoiled test panel, soiled panel (untreated), and on the soiled panel after scrubbing with a straight-line washability apparatus. Results can be either a visual rating or calculated as a percentage of soil removed.

6.2 *Materials:*

6.2.1 *Super Soil® Brand Potting Soil*[4], from local store,

6.2.2 *Palmitic Acid,* reagent grade,

6.2.3 *Stearic Acid,* powder, triple pressed,

6.2.4 *Coconut Oil,*

6.2.5 *Paraffin Wax,*

6.2.6 *Sperm Wax,*

6.2.7 *Olive Oil,*

6.2.8 *Squalene,*

6.2.9 *Cholesterol,*

6.2.10 *Oleic Acid,*

6.2.11 *Linoleic Acid,* purified,

6.2.12 *Sodium Stearate,* technical grade,

6.2.13 *Ferric Chloride,* hexahydrate, $FeCl_3 \cdot 6H_2O$,

6.2.14 *Calcium Stearate,* technical grade,

6.2.15 *Magnesium Stearate,*

6.2.16 *Carbon Lampblack,*

6.2.17 *Distilled Water,* and

6.2.18 *Synthetic Hard Water,* 100 ppm hardness, as $CaCO_3$, 3:1 Ca^{+2}:Mg^{+2}.

6.3 *Equipment:*

6.3.1 *Balance,*

6.3.2 *Stirplate/Hotplate,* two,

6.3.3 *Magnetic Stirbars,* two,

6.3.4 *Beakers,* 4000 mL, 2000 mL, 1000 mL, 600 mL (metal), and 500 mL,

6.3.5 *Forced Draft Oven,* capable of 110 ± 5°C,

6.3.6 *Buchner Funnel,* 500 mL,

6.3.7 *Vacuum Flask,* 2000 mL,

6.3.8 *Filter Paper,* Whatman 1,

6.3.9 *Separatory Funnel,* 500 mL,

6.3.10 *Ring Stand and Ring,* to fit separatory funnel,

6.3.11 *Mortar and Pestle,*

6.3.12 *Sieve,* 14 mesh,

6.3.13 *Ball Mill and Ceramic Jar,* with 1-in. diameter ceramic cylinders,

6.3.14 *Cookie Sheet,*

6.3.15 *Replacement Mini Trimmer Paint Pads,* two,

6.3.16 *Refrigeration,* for storage, 35°F (1.67°C), and

6.3.17 *Plastic Disposable Pipets.*

6.4 *Procedure:*

Pre-mix No. 1 Preparation

6.4.1 *Synthetic Sebum Soil*[5]:

6.4.1.1 Prepare synthetic sebum soil using the following constituents:

Constituent	Weight %
Palmitic acid	10.0
Stearic acid	5.0
Coconut oil	15.0
Paraffin wax	10.0
Sperm wax	15.0
Olive oil	20.0
Squalene	5.0
Cholesterol	5.0
Oleic acid	10.0
Linoleic acid	5.0
Total	100.0

6.4.1.2 Weigh the oils out into one beaker. Then weigh the powders out into another beaker. Weigh the paraffin wax and sperm wax into a third beaker.

6.4.1.3 Warm the oils on a hot plate, but do not allow mixture temperature to exceed 54°C. Add powders and stir until dissolved.

6.4.1.4 Add the paraffin wax and sperm wax and stir until all ingredients are dissolved.

[4] (Since 1954); a subsidiary of The Scotts Miracle-Gro Company, 14111 Scotts Lawn Rd., Marysville, OH 43041.

[5] Spanglers Sebum is commercially available and has been found suitable for this purpose. Scientific Services S/D, Inc., 42 Main St., P.O. Box 778, Sparrow Bush,NY 12780.

6.4.1.5 Pour the sebum into a glass jar and refrigerate at 35°F (1.67°C). The sebum may be stored at 35°F (1.67°C) for up to six months.

6.4.2 *Iron Stearate Preparation*—Iron stearate is not commercially available and must, therefore, be prepared in the lab. The following reaction is used to prepare iron stearate:

$$3\ \text{Na Stearate} + 1\ FeCl_3 \cdot 6H_2O \rightarrow 3\ NaCl + 1\ \text{Fe Stearate}_3 \quad (1)$$

6.4.2.1 Dissolve 15 g of $FeCl_3 \cdot 6H_2O$ in 285 g of distilled water. Pour solution into a 500-mL separatory funnel. Set aside until needed.

6.4.2.2 Add 40.5 g of sodium stearate to 1960 g of distilled water in a 4000-mL beaker. Stir mixture and heat to 60 to 80°C until sodium stearate is completely dissolved.

6.4.2.3 After the sodium stearate has dissolved, add the ferric chloride solution (from 6.4.2.1) slowly. Iron stearate will immediately begin to precipitate. After the addition of the ferric chloride solution has been completed, stir for an additional 5 min.

6.4.2.4 Remove beaker from heat and allow the mixture to cool.

6.4.2.5 When the temperature has dropped to 30 to 35°C, filter the mixture through Whatman 1 filter paper using a large Buchner funnel.

6.4.2.6 Wash the precipitate with distilled water (3 by 500 mL) to remove any remaining unreacted ferric chloride.

6.4.2.7 Allow the iron stearate to dry at room temperature to constant weight (approximately three days).

6.4.2.8 Iron stearate can be stored in a closed container for up to one year.

6.4.3 *Stearate Premix Preparation:*

6.4.3.1 Prepare stearate premix preparation using the following constituents in the order detailed as follows:

Constituent	Weight, g
Calcium stearate	53.0
Magnesium stearate	26.0
Iron stearate	21.0
Total	100.0

6.4.3.2 Combine the three stearates and pulverize with a mortar and pestle until homogeneous.

6.4.3.3 Stearate premix can be stored in a closed container for up to one year.

6.4.4 *SuperSoil® Brand Potting Soil Preparation:*

6.4.4.1 Remove the large wood chunks, vermiculite, and styrofoam particles from the potting soil with a large 14-mesh sieve.

6.4.4.2 Spread 1000 to 1500 g of the sieved potting soil over a cookie sheet and dry in a forced-draft oven at 110°C for 16 to 24 h until moisture is completely removed.

6.4.4.3 Fill the Ball Mill jar halfway with the ceramic cylinders. Place the sieved soil in the jar. Clamp the lid securely onto the jar and run the Ball Mill for a minimum of 4 h. The SuperSoil® dirt is now ready for use.

6.4.4.4 The SuperSoil® dirt may be stored in a closed container for up to one year.

6.4.5 *Final Premix No. 1 Preparation:*

6.4.5.1 *Composition:*

Constituent	Target Weight, g
Synthetic sebum soil	4.5
Stearate premix	3.0
Carbon black	0.6
SuperSoil® dirt	1.5
Total	9.6

6.4.5.2 Melt the sebum in a 600-mL metal beaker in a warm water bath at 65 to 70°C. In order to ensure homogeneity of the sebum, the mixture in the jar needs to be completely melted prior to sampling.

6.4.5.3 Add the remaining ingredients in the following order: stearate premix, carbon black, and SuperSoil® dirt. Mix after each addition until homogeneous.

6.4.5.4 Once the addition is complete, place a stirbar in the beaker and begin stirring at medium speed. Maintain the temperature of the mixture at 65 to 70°C.

Premix No. 2 Preparation

6.4.6 *Sodium Stearate Solution:*

6.4.6.1 Prepare sodium stearate solution as follows:

Constituent	Target Weight, g
Sodium stearate	40.29
Distilled water	259.71
Total	300.00

6.4.6.2 Add sodium stearate to distilled water.

6.4.6.3 Heat mixture to 75°C and stir until sodium stearate is dissolved and solution is clear. This can take up to 30 min to completely dissolve the sodium stearate.

6.4.6.4 Maintain temperature of solution at 75°C.

6.5 *Final Soil Preparation:*

6.5.1 Add Premix No. 2 to Premix No. 1 in 20-g increments until all of Premix No. 2 has been added. Stir (using stirplate) between each 20-g addition. Premix No. 1 and Premix No. 2 are heated throughout the addition procedure.

6.5.2 Stir at high speed with continued heating to obtain a homogeneous mixture. Soil color should be charcoal gray.

6.5.3 Maintain a soil temperature of 75 to 80°C throughout the soil application.

6.5.4 This soil mixture can be stored in a closed container at 35°F for up to 1 year.

7. Preparation of Substrate

7.1 Wash tiles in a laboratory dishwasher using an industrial-strength dishwashing detergent.

7.2 Air-dry tiles for 24 h. For best drying, position tiles such that air flows freely across all surfaces.

7.3 Preheat oven to 70 to 80°C.

7.4 Place tiles on an oven tray and heat for at least 1 h before applying the soil. Soil enough tiles to run eight replicates per test product plus three extra for cycle determination.

8. Soil Application

8.1 *Apparatus:*

8.1.1 *Soil Applicator,* mini trimmer paint applicator[6] (3.175 by 6.985 cm).

8.1.2 *Test Substrate,* white ceramic glazed wall tiles (10.795 by 10.795 cm).

8.2 *Tile Preparation:*

8.2.1 Remove the tray of tiles from the oven immediately prior to soil application. Tiles can be soiled while hot or allowed to cool to room temperature.

8.2.2 Use the mini trimmer paint pad[6] and applicator to apply a single layer of hot soil down the center of a hot tile. Dip the pad in the soil as it is stirring and squeeze out the excess by pressing pad against the side of the beaker. It is easiest to soil an entire row of tiles with one stroke of the applicator (line the tiles up in a row so that the tile edges are touching each other and apply the soil with a broad stroke of the applicator).

8.2.3 Place the soiled tiles back in the oven and heat at 70 to 80°C for 1 h.

8.2.4 Remove the soiled tiles from the oven and cool overnight prior to testing. Visually inspect tiles for uniformity prior to use. A standard initial reflectance reading is recommended for use to ensure application uniformity, minimum tile variability, and optimum sensitivity. For reference, a Photovolt reflectometer[7] should yield an initial Y reflectance reading of 35 ± 5 units, although a different reflectometer or colorimeter may yield different readings. The range with a colorimeter using the XYZ scale and daylight setting ($D65/10^0$) is Y(B)40.0 ± 5.6.

8.2.5 Tiles may be used for testing up to three months after preparation when stored at 35°F (1.67°C).

8.2.6 Randomize tiles prior to cleaning process.

9. Cleaning Procedure

9.1 *Apparatus:*

9.1.1 *Test Sponge,* cellulose sponges, fine pore (damp dimensions: 1.588 by 7.30 by 11.748 cm).

9.1.2 *Scrubbing Apparatus,* straight-line washability apparatus.

9.1.3 *Test Sponge Holder,* a standard sponge holder for the straight-line washability apparatus unit, or a flat metal plate (5.5 by 3 in.) with spikes on bottom have been found suitable.

9.1.4 *Tile Template,* Plexiglass tile holder, cut to fit into tray on washability apparatus (17.145 by 55.245 by 0.635 cm). The holder should have a hole (11.43 by 11.43 cm) in the center to hold tiles. Unsoiled tiles may also be used to hold the soiled tiles in place.

9.2 *Cleaning Test:*

9.2.1 *Sponge Preparation:*

9.2.1.1 Sponges, as received, contain small amounts of surfactants and thus need to be cleaned prior to using. Wash sponges in a washing machine using warm water and spin dry. Dry the sponges in a clothes dryer until they are completely dry. Discard sponges with surface imperfections, uneven thickness, or other obvious deformities. If sponges have been washed by the manufacturer, then just air dry.

9.2.1.2 Prior to applying product, tare a clean, dry sponge. Submerge the sponge in synthetic hard water (100 ppm as $CaCO_3$, 3:1 Ca^{+2}:Mg^{+2}) and squeeze out all but 17.5± 0.5 g of water.

9.2.2 *Application of Test Product*—Test product dilutions should be prepared from synthetic hard water (100 ppm as $CaCO_3$, 3:1 Ca^{+2}:Mg^{+2}) at ambient temperature. Dilutions used should be prepared fresh for each test, and should be made at the manufacturer's recommended cleaning dilution.

9.2.2.1 *Dilutable Products*—Dilutions should be made on a weight/weight basis (for example, a 1-oz to 1-gal dilution would be made by diluting 1 g product with 127 g water). For dilutable products, the usage dilutions should be according to manufacturer's label instructions for **heavy** soil removal. Evenly apply between 10 to 20 mL of the diluted product to one face of the sponge with a plastic, disposable pipet, covering the face as completely as possible.

9.2.2.2 *Nondilutable Products:*

(a) Liquid Cleaners—Evenly apply between 10 and 20 mL of product to one face of the sponge with a plastic, disposable pipet, covering the face as completely as possible.

(b) Liquid Abrasives—Apply between 1 and 5 g in a band across the middle of the sponge perpendicular to its long edge.

(c) Powdered Abrasives—Apply between 1 and 5 g of the powder across the middle of the sponge perpendicular to its long edge.

9.2.2.3 *Spray- or Foam-on Products*—When the label directions specify application to the soiled surface, evenly apply 2–3 g of product to the surface to be cleaned. Let stand 1 min (or other time as specified).

9.2.3 *Cleaning Soiled Panels*—Set scrubbing speed of the washability apparatus at 40 cycles/min. Place the plexiglass tile holder in the washability apparatus. Place a soiled tile in the hole in the center of the tile holder. Apply product to sponge as indicated in 9.1.3. Do not apply products described in 9.2.2.3 to sponge. Set the test apparatus at the predetermined number of cycles, established in the procedure described in 9.2.4. The sponge holder head should be positioned 4 in. to the left of the tile at beginning of first cycle. Remove the tile from the holder and rinse under a light stream of 25°C tap water. Let air dry prior to evaluating cleaning performance.

9.2.4 *Establishing a Standard Number of Cycles for Test Product Evaluation*—Place tiles in the washability apparatus with the line of soil on the tile running perpendicular to the cleaning direction of the scrubbing apparatus. Using extra tiles, run standard products to determine product performance profiles. It is suggested that the standard reference products remove approximately 75 % of the soil, in order to allow for maximum product differentiation. Identify the cycle number at which maximum differences in product performance are demonstrated. Run all test products with this predetermined standard number of cycles.

[6] Shur-Line® trim & touch-up pad or equivalent: A Newell Rubbermaid Company, St. Francis, WI 53235.

[7] Photovolt/ Division of UMM Electronics, 6911 Hilldale Ct., Indianapolis, IN 46250–2062.

10. Visual Evaluation

10.1 The treated tiles are displayed on a flat, neutral colored (gray) non-glare finished surface under lights simulating standard daylight.[8]

10.2 Tiles, within a group, are randomized for grading in order to minimize treatment/judgement bias.

10.3 Soil removal is visually evaluated by a minimum of eight independent judges to the nearest 0.5 rating. A clean tile and a soiled tile are used as references. Soil removal is rated as follows:

Rating	Description
1	No or very little soil removed.
2	Approximately 25 % soil removed.
3	Approximately 50 % soil removed.
4	Approximately 75 % soil removed.
5	Virtually all soil removed.

11. Instrumental Evaluation

11.1 Measure the reflectance of the reference and treated tiles with a Photovolt reflectometer.[7]

[8] A simulated daylight source is intended (7500 ± 300 K). Refer to Practice D1729 and Test Method D2960.

11.2 The percentage of cleaning efficacy of the test products is calculated using the formula:

$$\%\ \text{cleaning efficacy} = \frac{(R^c - R^s)}{(R^o - R^s)} \times 100 \quad (2)$$

where:

R^c = cleaned reflectance,
R^o = original reflectance, and
R^s = soiled reflectance.

11.2.1 *Calculation of the Mean and the Standard Deviation*—The mean of the values should be calculated by the following formula:

$$\bar{X} = \left(\sum X/n\right) \quad (3)$$

where:

$\bar{X}$ = mean (average) cleaning efficacy,
X = cleaning efficacy (from 10.2), and
n = number of readings.

11.3 Statistical analysis of variance can establish significant differences between the test treatments.

12. Keywords

12.1 abrasive cleaners; bathroom soil; ceramic tile cleaners; hard-surface cleaners; soap scum

SUMMARY OF CHANGES

Subcommittee D12.16 has identified the location of selected changes to this standard since the last issue (D5343-06) that may impact the use of this standard.

(1) Added to 6.1 Summary of Method – "The soiled tiles can be stored for up to three months when stored at 35°F.
(2) In 6.2.1 and throughout: SuperSoil® Brand Potting Soil was added to replace Super Mix potting soil.
(3) Added footnote references to 6.4.1, 8.1.1 and 8.2.2:
(4) Added to 6.5.4: This soil mixture can be stored in a closed container at 35°F for up to 1 year.
(5) Added to 8.2.1: Tiles can be soiled while hot or allowed to cool to room temperature.
(6) Added to 8.2.5: Tiles may be used for testing up to three months after preparation when stored at 35°F (1.67°C).