An Optimized Hard Surface Cleaning Test

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Abstract

Preliminary investigation of several test procedures resulted in the selection of the Liss-Hilton reflectance method. A white vinyl tile substrate was selected and the test method optimized at cleaning efficiencies of approximately 30% for water and 80% for selected detergent compositions. Within-series and between-series variations, in general, met a required 2% maximum. Between-laboratories data were slightly more variable. Utilizing the corresponding confidence limit factor, as few as three test panels per detergent may be used to compare detergent cleaning efficiencies.

Introduction

Hard-surface cleaners are used in large volume, and though several test procedures have been used, no entirely satisfactory test for measuring cleaning efficiency appears to exist. An early federal specification, P-C-431 (2), was designed to evaluate syndets for cleaning painted surfaces. With essentially the same soil, but also applying the method to linoleum, Harris and co-workers (4) demonstrated that the method was applicable to both substrates. A federal specification (P-D-220a) covering a general-purpose detergent (1) is currently used in procuring detergent materials, and requires the painted-surface substrate. Liss and Hilton (5), using much the same soil as those cited above, selected white vinyl tile as the substrate, and their data indicated improved test reproducibility.

The foregoing citations indicate the state of the art, but before selecting one of these procedures for optimization, other approaches to the problems of soil and substrate were considered. Harris et al. (3) applied radiotagged soil to metal surfaces and radioactive soil was also used by Shelberg et al. (7). While these soils and this technique are quite sensitive and reliable, none has attained the status of a control method. When considering other approaches to the problem of a standardized soil removal test, the salient factors are need for a reproducible and practical substrate, and a tracer material which can quantitatively indicate the degree of soil removal. In the foregoing discussion, radiotracers have not proved adaptable, whereas much effort has gone into examination of colored tracers whose removal is measured by reflectance, much as visual inspection is used on a practical basis. Other measurement techniques such as extraction of the tracer after washing followed by spectrophotometry or other advanced techniques have not offered the simplicity and practicability necessary for control testing. Various types of chromatography, atomic absorption, NMR, mass spectrometry or use of the electron scan microscope are possibilities of a means for measuring a tracer, but for various reasons none has been considered wholly applicable to the problem.

The objectives of this project were to show the steps taken in the vinyl tile method optimization to attain intra- and interlaboratory repeatability and reproducibility.

TABLE I
Detergent Formulations Used for Optimization

Component	Weight per cent	
	Detergent 1	Detergent 2
Santomerse 85[R] (alkylbenzene sulfonate)[a]	23.5	......
Sterox 66L[R] (nonionic ethoxylate)[a]	......	10.0
Tripolyphosphate	40.0	40.0
Sodium metasilicate (pentahydrate)	7.0	7.0
Sodium sulfate (anhydrous)	29.5	43.0

[a] Monsanto Company.

Materials and Methods

Test Requirements

The following test requirements were established to provide a satisfactory basis for test usage: (a) Utilization of a straight-line washability apparatus and a suitable flat substrate. (b) Development of a soil-substrate system wherein distilled water removed ca. 30% of the soil and ca. 80% is removed by either of two detergent solutions at 0.5 wt% concentration. (c) Goals were established of not greater than 5% removal variation within a test series, and not greater than 2% between test series. Between-laboratory variation was desired at a level not greater than 2%. (d) The number of replicate test panels to achieve the goals was to be established.

Test Materials

To minimize effort, a white vinyl tile was used for preliminary investigation. The Liss and Hilton test method was used initially, with improvements in procedure and soil preparation being the major features of optimization. The optimized method is included and improvements in the original method are indicated in the text.

The detergent formulations used for optimization are found in Table I.

Soil

Table III shows the soil formulations used, in which it is apparent that only minor variations exist.

Preliminary tests were made with the P-D-220a soil to determine whether, on the vinyl tile, the soil was susceptible to variation in temperature and time of soil-cure. Not only did moderate changes in temperature or time of cure have considerable effect, but the soiling mixture itself aged excessively. For these reasons this soil was abandoned in favor of the other two, which are essentially of the same general composition, varying only in solvent and amounts.

The Liss-Hilton soil was chosen for further investigation because it had been more recently

TABLE II
Per Cent Cleaning Efficiency

Tiles	Detergent 1	Detergent 2
Corlon	100	93.8
Source 2	97.9	97.0
Amtico	95.3	88.3

TABLE III

Tile Soil Formulations

Material	Liss-Hilton (parts)	Harris (parts)	P-D-220a
Metallic brown	20	20	20 g
Graphite	....	....	1 g
Carbon tetrachloride	....	20	....
Kerosene	12	20	....
Stoddard solvent	12	....	....
Mineral spirits	....	....	40 ml
Liquid petrolatum	1	....	2.5 g
Nujol	....	1	....
Lubricating oil (SAE 10)	1	....	1 ml
Lubricating oil (SAE 60)	....	1	....
Hydrogenated vegetable shortening	1	....	1 g
Hydrogenated vegetable oil	....	1	....

TABLE IV

Multiple vs. Single Sponge Usage Cleaning Efficiency[a]

Value	Detergent 1			Detergent 2		
	Fresh sponge each panel	3 Sponges re-used	1 Sponge re-used	Fresh sponge each panel	3 Sponges re-used	1 Sponge re-used
$\bar{X}$	78.7	78.2	78.6	71.6	72.2	75.0
R	10.6	4.7	4.7	12.0	6.7	5.3
σ	3.7	1.9	1.6	3.9	2.0	2.1
V	4.7	2.4	2.1	5.4	2.8	2.7
N	9	9	9	9	9	9

[a] Liss-Hilton soil; 0.008 in.; cured 60 min @ 110 C.

evaluated, and was of better physical consistency for application by a film casting knife. Brush application is difficult to reproduce, hence it was not further investigated.

The soil formulation was aged 5, 12 and 25 days, and the cleaning efficiency for the three soil batches was found to vary from 91.4% to 93.7% for Detergent 1, and 87.8% to 90.4% for Detergent 2. These tests showed quite adequate storage characteristics, but a cleaning efficiency (CE) of 80% was desired. For cleaning efficiency, see section on Hard Surface Cleaning Test. This meant variation in time and temperature of cure or initial soil thickness or both. Time of cure was varied in ¼ hr intervals up to 2 hr, temperature levels were either 80 or 100 C, and initial soil film thickness was either 0.004 or 0.008 in. Both detergents at 0.5 wt % solution concentrations were used for CE determination. At cure conditions of 1 hr at 100 C and initial soil thickness of 0.008 in., the Detergent 1 variation for six panels were 77.3% to 81.6%, and for Detergent 2, 72.0% to 80.0%. Since an 80% CE level was the goal, these conditions were used for subsequent tests. With deionized water under these soiling conditions, CE was on the order of 17% to 20%.

Two different supplies of metallic brown were tested and no significant differences in their usage was found.

There was no effect upon CE whether cured panels were used within the first 1 to 3 hr or up to 20 to 24 hr after preparation.

Effect of ±10 C variation in cure temperature at the 60 min cure time had no significant effect upon CE.

Substrate

Liss and Hilton had successfully used Armstrong Corlon white vinyl tile, and portions of the exploratory work on the soil were executed with this tile, now no longer available. Other sources were sought; American Biltrite Rubber Company's Amtico VP-11 Plain White vinyl flooring and another, Source 2 tile, were obtained.

Preliminary testing indicated that these tile panels reacted differently to a single set of application and cure conditions. Deionized water was used with the three tile supplies and Corlon showed an average CE of 72.7% (six panels), Source 2 an average CE of 81.6%, and Amtico an average of 40.4%. Table II gives the cleaning efficiencies obtained when the tiles were washed with the two detergents. Under these application conditions, and realizing that only Amtico tile approached the 30% water-removal level, which provided a broad range between no-detergent and detergent-application, this tile was chosen for further testing. Reproducibility of tile from one supply lot to another will be discussed later. The optimum cure and application conditions were discussed under the Soil section.

Sponge Effect

Fine-pore viscose sponges are one of the test variables and their effect on CE by single usage through a test or by using individual sponges for each panel was determined. An estimation of this effect is shown in Table IV. It is apparent from these data that lower test variation occurs when fewer sponges are used in the test. To all intents and purposes, a single sponge is preferably to be used for a single test series.

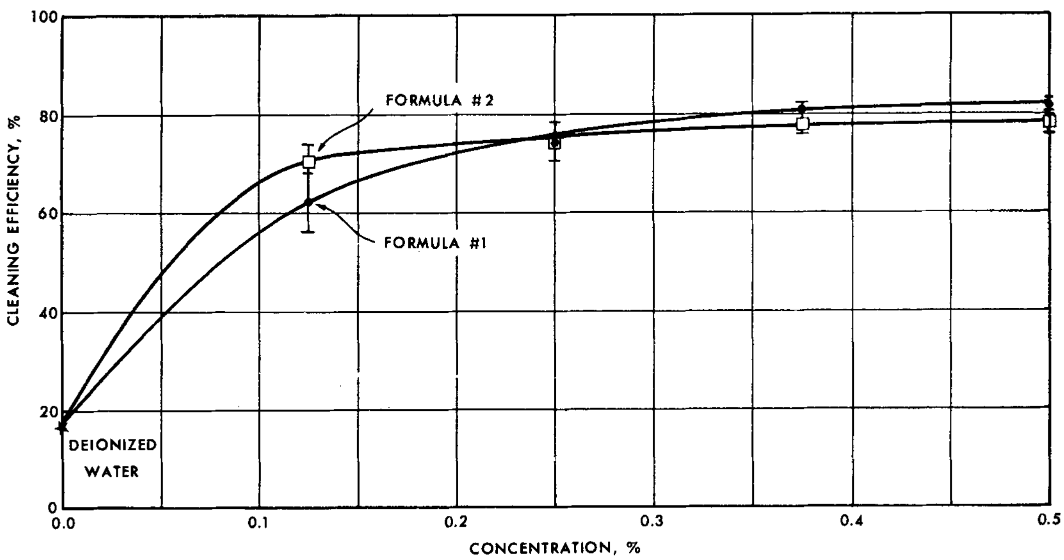

FIG. 1. Effect of detergent concentration.

Effect of Detergent Concentration

Tests were made with detergents at 0.5 wt % solution concentration (in 150 ppm hard water). Satisfactory CE levels were attained at this level, but in practical application, some of the products would be tested at less than 0.5% concentration. Consequently, tests of both detergents were made at 0, 0.125, 0.25, 0.375 and 0.5 wt % concentration levels. The curves in Figure 1 show the greatest apparent deviation at 0.125% concentration, but here, the 95% confidence levels overlap, hence no real difference exists. However, a real difference exists at 0.5%, with Detergent 1 giving greater CE. Detergent 1 at 0.125% is significantly lower than at 0.25%, indicating that its CE is affected by dilution. The 0.5% concentration level therefore appears applicable to these detergents.

Repeatability Between Tile Shipments

Before proceeding with an analysis of the repeatability data, a short summation of the statistical methods used is appropriate. It is expected that this wash test, like most effective detergent methods, will produce data at a 95% confidence level. While there are a number of statistical methods which can be applied to determine whether real differences can be shown at this level, one of the easiest to apply is that by Lord (6). Here the range, i.e., the difference between the highest and lowest of the values, is simply multiplied by a factor dependent upon sample size and confidence limit. Another approach is to apply a similar factor to the standard deviation of the test series. Both give identical results, but the latter is considerably more time-consuming.

Since the sample size chosen was nine, at the 95% confidence level, the factor (f_{95}) taken from Lord's Table 10 (6) was 0.334. Hence the range of any test series multiplied by this factor will give the 95% confidence limits (CL_{95}). To determine whether statistical differences exist between two test series, the following procedure is used: the difference between averages of the two series is calculated, and the CL_{95}'s for the two series are totaled. If the difference between the series averages is equal to, or greater than the sum of the CLs, the chances are 95 in 100 that a real difference exists between the two series.

Since four shipments of Amtico tile were secured for this work, and since each shipment appeared to be from different production lots, reproducibility of the tile was determined. Table V shows representative detergency values for four different boxes prepared and washed under identical conditions.

In analyzing these data, the following conclusions

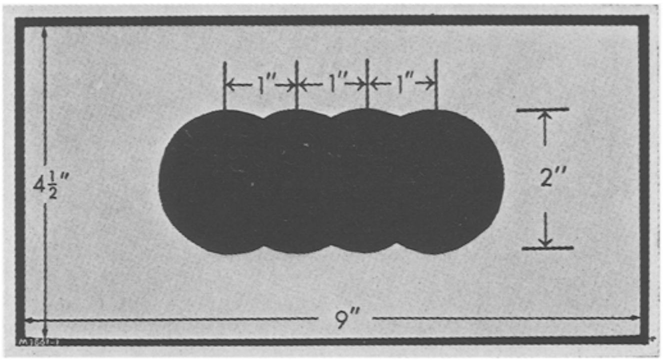

FIG. 2. Template dimensions.

may be drawn: Detergent 1, in all four cases, had statistically higher CE values than Detergent 2. Box 2 gave lower CE values for both detergents than the other three boxes which within themselves statistically showed no differences. These data show that differences between lots can occur, notwithstanding that the relative CE differences in the detergents are still maintained. It should be noted that for control purposes, a sufficient volume of test tile should be secured as a single lot.

Repeated tests of Box 2 gave the following order of CE values and their associated CL_{95} values for Detergent 1: 78.8, 4.2; 78.2, 1.6; 78.6, 1.6; 78.5, 2.2; and 77.1, 2.0. For Detergent 2 these values were: 71.6, 4.0; 72.2, 2.0; 75.0, 1.8; 72.1, 2.2; and 69.7, 4.0.

Test Replicates

A test procedure should produce a value at some predictable level of repeatability and reproducibility for a minimum expenditure of manpower. For a given procedure, the larger the number of replicate values, the greater the probability that a more precise mean value will be attained. However, dependent upon the purpose of the analysis and the required probability level, the labor required to produce a desired result may be minimized.

In this work, a sample of nine replicate panels was selected so that an analysis of the data could be made and shorter test series could be used. The controlling factor in these tests was the repeatability of the CE value as indicated by the range of values

TABLE V

Repeatability Between Tile Shipments
Amtico Tile
(Single sponge)

Value	Box 1	Box 2	Box 3	Box 4
Detergent 1				
$\bar{X}$	80.5	78.5	83.0	80.3
N	9	9	9	9
R	8.0	6.7	4.6	6.0
f_{95}	0.334	0.334	0.334	0.334
CL_{95}	2.7	2.2	1.5	2.0
Detergent 2				
$\bar{X}$	75.7	72.1	76.6	77.7
N	9	9	9	9
R	10.0	6.7	9.2	3.3
f_{95}	0.334	0.334	0.334	0.334
CL_{95}	3.3	2.2	3.1	1.1

TABLE VI

Test Sample Size

Size replicate	Value	Test value	f_{95}	Test value	Test value
Detergent 1					
9	$\bar{X}$	80.5			
	R	7.3			
	f_{95}	0.334			
	CL_{95}	2.4			
5	$\bar{X}$	81.3		79.9	
	R	4.6		6.7	
	f_{95}		0.613		
	CL_{95}	2.8		4.1	
3	$\bar{X}$	81.3		81.3	78.5
	R	4.6		1.4	6.7
	f_{95}		1.272		
	CL_{95}	5.9		1.8	8.5
Detergent 2					
9	$\bar{X}$	75.7			
	R	10.6			
	f_{95}	0.334			
	CL_{95}	3.5			
5	$\bar{X}$	74.1		77.3	
	R	7.3		4.0	
	f_{95}		0.613		
	CL_{95}	4.5		2.4	
3	$\bar{X}$	73.3		76.0	77.8
	R	7.3		1.3	4.0
	f_{95}		1.272		
	CL_{95}	9.3		1.7	5.1

obtained. However, at the 95% probability level, the factor to be applied to the test series range increases markedly the fewer the panels in a series. Specifically, the factor varies as follows: nine panels, 0.334; five panels, 0.613; and for three panels, the practical minimum, the value is 1.272. The factor increases in the ratio 1:2:4, so that if fewer panels are tested a larger CL_{95} value may be expected.

To test the effect of reducing sample size, sets of nine panels were arbitrarily divided into two sets of five, with the fifth panel of the nine-panel series in each set: Further, the nine panels were reduced to three series of three panels each. Table VI is an example of several series so analyzed. As could have been expected, variation in the CL_{95} values is greater for the three-panel tests, but increase in the CL value was not necessarily in the ratio of 1:2:4.

It becomes evident that as long as the test has been polished to the point where test variation has been minimized, comparisons between samples can be made with the applicable CL values, and that three panels can be used as well as five or nine. What may be marginally lost in a degree of certainty is largely gained in the ability to perform more tests in the same time period.

The preferred test method developed, as a result of the foregoing tests, is to be found in the following section.

Hard Surface Cleaning Test

Scope. This method covers the determination of the cleaning efficiency by detergent solutions in removal of a synthetic soil from a vinyl tile surface.

Summary of Method. The method consists of the removal of a synthetic soil from a white vinyl surface using a washability apparatus followed by reflectance measurement for calculation of cleaning efficiency.

Apparatus. Washability Apparatus (Gardner Laboratory, Inc., Bethesda, Md.): This consists essentially of an electric motor mounted on a metal plate and a mechanism through which the motor imparts a reciprocating motion to a sponge held in a metal box. The sponge box is 3¾ in. long, 2⅞ in. wide and 1 in. deep fitted with a ½ in. plastic insert and is set in a narrow lipped stainless steel pan in which a test panel is centrally located. The sponge of type 1 of Federal Specification L-S-626, preshrunk, and cut to a dry size to fit the sponge box with a wet thickness of 1 in. The total weight of the box and dry sponge shall be 1 lb. ±½ oz.

Photometer (Model 610, Photovolt Corp., New York): A photometer and search unit fitted with an amber tristimulus filter is standardized between series of measurements using a ca. 80% reflectance standard plaque.

Film Applicator: A film applicator should give an opening 3 in. wide and of variable gate heights.

Template: A template is used for reflectance measurements for snug contact with the surface to be measured as shown in Figure 2.

Forced Draft Oven: The oven should be capable of operating at 212 ± 0.1 F.

Reagents and Materials. Vinyl tile (American Biltrite Rubber Co., Inc., Trenton, N.J.): Amitco VP-11 plain white tile 9 × 9 × ⅛ in.

Standard Detergent: Dodecylbenzene sodium sulfonate (Santomerse 85, Monsanto Co., St. Louis, Mo.), 23.5 wt%; sodium tripolyphosphate, granular, 40.0 wt%; sodium metasilicate pentahydrate, ASTM D 537, 7.0 wt%; sodium sulfate, anhydrous (ACS), 29.5 wt%.

Soil Materials: Metallic brown (Bureau of Standards Sample 307), 20 parts (by weight); odorless kerosene (Fed. Spec. UV-K-211c), 12 parts; Stoddard solvent (Fed. Spec. P-S-661B, Type 1), 12 parts; white mineral oil (USP), 1 part; lubricating oil (SAE-10, ML), 1 part; and hydrogenated vegetable shortening (Crisco, Procter and Gamble Co.), 1 part.

Combine oils and shortening and heat to 106 ± 2 F to melt. Add half of the kerosene then mix in metallic brown pigment. When smooth, add remaining kerosene and Stoddard solvent and complete mixing.

Synthetic Hard Water: Accurately weigh 0.132 g/liter $CaCl_2 \cdot 2H_2O$ (CP) and 0.1475 g/liter $MgSO_4 \cdot 7H_2O$ (CP) and dissolve them in a small portion of distilled or deionized water, transfer to a liter flask and make up to volume.

Soiled Tile Preparation. Cut the 9 in. tiles into halves and sponge scrub by hand with a 0.5 wt% solution of dodecylbenzene sodium sulfonate (85%) in tap water, rinse, drain, and dry with absorbent paper towelling, then oven dry 15 min at 212 F. Cool on a flat surface to room temperature. Measure reflectance before soiling.

Evenly soil panels with about 5 g of homogeneous soiling mixture in a 3 in. width by 0.008 in. in thickness with a blade applicator. Air dry panels at least 30 but not more than 60 min, then cure panels on a smooth surface in the oven at 212 ± 5 F for 60 min. Remove panels from oven and cool to room temperature, using the panels within 24 hr.

Washing Procedure. Prepare 0.5 wt% solutions of detergents in the synthetic hard water or test at a recommended concentration.

Soak soiled panels for 60 sec in wash solution sufficient to cover the entire panel, wet sponge with 77 ± 5 F tap water and squeeze damp dry. Add 50 ml of detergent solution to sponge and insert sponge in box. Center test panel in washability pan with two properly sized pieces of tile. Start apparatus at one stroke per second (one cycle back and forth is two strokes), while dripping detergent solution from a pipet onto test panel center at a rate of 12 ml of solution during the wash period of 100 strokes. Stop apparatus, remove and rinse test panel under light stream of 77 ± 5 F tap water. Drain and replace panel in apparatus in the reverse direction. Rinse sponge in 77 ± 5 F tap water, squeeze damp dry, add 50 ml of detergent solution to unused side, replace sponge in box and repeat 100-stroke wash cycle as before. Rinse panel and air dry.

Cleaning Efficiency Measurement. Using a photometer and search unit equipped with tristimulus amber filter, measure reflectance of the surface of the test panel before soiling and after washing using a vinyl tile template of the dimensions given in Figure 2. The four readings for each panel are measured to the nearest 0.5%, and averaged. Between panels the search unit is rested on a standard white plaque of approximately 80% reflectance and the instrument checked before each series of readings.

Calculation. Calculate the percentage cleaning efficiency as follows:

$$\text{Per cent cleaning efficiency} = \frac{R_2}{R_1} \times 100$$

TABLE VII
Comparison of Commercial Products

Sample	Amount used	$\bar{X}$	R	N	f_{95}	CL_{95}
Detergent 1	0.5 wt%	87.2	0.7	4	0.813	0.6
A[a]	0.5 vol%	80.6	5.2	5	0.613	3.2
B	1.0 vol%	27.2	4.6	5	0.613	2.8
C	0.375 wt%	83.7	6.9	5	0.613	4.2
D	1.0 vol%	85.9	5.2	5	0.613	3.2
E	0.75 wt%	67.7	7.3	5	0.613	4.5
F	0.88 vol %	35.1	14.3	5	0.613	8.8

[a] At recommended use concentrations.

where R_1 = reflectance of unsoiled, unwashed panel; R_2 = reflectance of soiled, detergent washed panel. Make triplicate determinations of cleaning efficiency by the above procedure and average the results obtained.

Comparison of Commercial Products

Table VII shows the data resulting from tests of six commercial products compared with Detergent 1. These data were obtained with yet another shipment of tile, and the tile-Detergent 1 CE values were somewhat greater than with other procurement. This fortifies the recommendation that a reasonably large volume of tile be procured for extended test work.

The commercial detergents were used at their recommended usage levels, and it is apparent that more than half of them failed to approach Detergent 1 in cleaning efficiency, i.e., only Samples C and D statistically matched the cleaning efficiency of the comparison detergent.

Between-Laboratories Tests

A round-robin test was established with four participating laboratories, all indicated as versed in this type of performance test. Each participant was furnished with identical test materials and panels. Furthermore the participants were directed to perform a short test series to familiarize themselves with the technique. A series of nine panels was to be used for each of Detergents 1 and 2, and the complete test was to be repeated the following day, thus giving two complete test series. The panels for the tests were all from the same shipment to obviate this variable.

Since our use of the test had shown no real difficulty in operation though reduction in test variability occurred with time, it was hoped that the other laboratories would have the same experience. That the test is not to be taken lightly was evidenced by one laboratory which used an inexperienced operator whose values were completely invalid, but when one experienced in the method was used, the values very closely matched our own. Another laboratory failed to provide valid data even after three separate trials, though later data more nearly approached normal values. And one highly pleasant experience was that one laboratory matched our results the first time through, indicating, along with another of the laboratories, that careful attention to test detail can give reproducible results.

The data are shown in Table VIII. (Laboratory A was Monsanto Research Corporation, Dayton Laboratory.) Upon return of the tile, they were reflectance-measured for comparison with values obtained by the cooperating laboratories. These measurements suggested this as a factor which could cause apparent differences in absolute CE values, but when a comparison detergent is used, adequate correction should then result. This is not the first case in which differences in reflectance values have been experienced in round-robin tests, even though standardized reflectance plaques are used.

Analysis of the Laboratory A readings for the four laboratories indicates greater variability for Detergent 1 than Detergent 2 and the mean values for the latter are all within the confidence limits. For Detergent 1, with the exception of Laboratory A, Test 1 and Laboratory D, the CE values obtained were within the confidence limits. It is believed that with further experience the laboratories could reduce their range variabilities, and that the mean values between laboratories would more nearly coincide. The confidence limits for these inter-laboratory tests closely approach the desired 2% between-laboratories variation.

Test-Method Controls

Aside from the clearly specified conditions outlined in the procedure, several general variables should be controlled. These are the following: (a) A sufficient supply of tile from one production lot should be procured for an extended series of tests. (b) A sufficient supply of the soil components should likewise be procured. (c) The chemicals for a standard detergent formulation, preferably Detergent 1, should be procured in adequate supply. (d) New supplies of

TABLE VIII
Between-Laboratories Tests of Nine Replicate Panels

Value	Laboratory A—Box 3			Laboratory B			Laboratory C	Laboratory D
	Test 1		Test 2	Test 1		Test 2		
Detergent 1								
Laboratory A readings								
$\bar{X}$	85.6		83.0	81.4		81.2	82.2	87.6
R	6.0		4.6	6.6		7.8	8.1	8.8
f_{95}		0.334			0.334		0.334	0.499[a]
CL_{95}	2.0		1.5	2.2		2.6	2.7	4.4
Individual laboratory readings								
$\bar{X}$	85.6		83.0	83.9		88.1	82.8	93.2
R	6.0		4.6	7.1		4.6	8.9	8.3
f_{95}		0.334			0.334		0.334	0.499[a]
CL_{95}	2.0		1.5	2.4		1.5	3.0	4.2
Detergent 2								
Laboratory A readings								
$\bar{X}$	79.1		76.6	79.5		76.9	78.7	78.9
R	7.9		9.2	12.5		10.5	7.8	16.3
f_{95}		0.334			0.334		0.334	0.499[a]
CL_{95}	2.6		3.1	4.2		3.5	2.6	8.1
Individual laboratory readings								
$\bar{X}$	79.1		76.6	81.8		79.7	79.2	83.6
R	7.9		9.2	10.3		11.4	7.4	15.1
f_{95}		0.334			0.334		0.334	0.499[a]
CL_{95}	2.6		3.1	3.4		3.8	2.5	7.5

[a] 6 panels.

any kind or a new test operator should be checked out against the selected standard detergent. (e) A standard detergent should be tested at least occasionally.

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