glyceride composition of the tallows, as suggested by Hilditch and collaborators or by Longenecker. The increased melting points produced on interesterification are explained on the basis of the GS_3 contents of these fats as determined by Riemenschneider and collaborators. Both the increased melting points and increased micropenetration values are shown to be readily explainable on the basis of the glyceride type composition assigned to these tallows by the writer.

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Detergency Evaluation. III. Adjustment of Terg-O-Tometer and Launderometer Wash Test Methods to Produce Comparable Soil Removal Data

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PART I (1) of this series was concerned with the evaluation of several wash test methods, using one standardly soiled test fabric. Part II (2) of the series described the results obtained, using three standardized wash test methods with four different soiled test fabrics. The present paper presents the results obtained in an attempt to adjust the Terg-O-Tometer wash method to provide soil removal data and detergent comparison results equivalent to the Launderometer wash method using four 10-minute washes.

Bacon and Smith (3) demonstrated that one of the major factors in the removal of soil was the amount of mechanical work applied during the washing operation. Their work was done in the Launderometer, varying the energy applied by changing the number of stainless steel balls used with the wash load. The soil removal characteristics were shown to be directly proportional to the amount of mechanical work done.

The work to be reported here has a very practical application because many laboratories use the Launderometer while others use one or another of the machines recently designed to overcome certain specific shortcomings of those previously used. These machines were developed to simulate either actual washing principles, as with the Terg-O-Tometer, or to provide highly variable but controllable application of energy to the washing operation.

In most cooperative efforts to evaluate soiled fabrics or detergents much variation has been encountered in spite of effort to control all variables. This is attributed to a considerable extent to variation in technique. Part II of this series indicated that the Terg-O-Tometer might prove more valuable for interlaboratory testing because the technique involved is much less complicated than for the Launderometer methods.

The present work was undertaken as an attempt to develop a wash test method using the Terg-O-Tometer, which would essentially reproduce the degree of soil removal obtained by our Launderometer method, provide the same statistical comparison between detergents, reduce the amount of time required for the test in comparison with the Launderometer, and provide more data in the way of a larger number of replicate samples for improved test reproducibility.

Soiled Test Fabric

The soiled test fabric used was a carefully standardized product already described in detail (4). Briefly, standardization constituted maintenance of soil removal characteristics under specified wash test conditions using a standard detergent. Batches of soil were chosen which fall within narrower limits than those generally established for the statistical control limits.

Wash Test Methods

Launderometer. Four 10-minute washes. This method was described in considerable detail in Part I of these papers. Briefly it utilized the Launderometer, multiple washes (four, of 10 minutes each), 100 ml. of fresh solution for each wash, 10 rubber balls per jar, and two hand rinses.

Terg-O-Tometer Method. This method consisted of single washes of varying duration. The wash test load was adjusted to either 30 or 60 g. per liter, using 500 ml. of detergent solution. The load consisted of 5 swatches of standardized soil, 4″ x 6″ in size, trimmed to provide the required weight of fabric to volume of solution. After the wash of either 3, 5, 10, 20, or 40 minutes' duration, the swatches for any detergent in question were piled one on top of the other and wrung once through a hand wringer. They were then rinsed in water of the same hardness in the Terg-O-Tometer for five minutes (or 2 minutes with the 3-minute wash) at the same temperature as the wash, and then again wrung through the hand wringer, placed on an aluminum drying plate in a flat position, and oven dried.

The cycle rate was maintained as in previous tests at 144 per minute. The angle of rotation of the agitators was maintained as before at 345-350°.

Wash Test Conditions.

Water hardness—50 ppm.
Detergent solution—0.2%
Temperature—140°F.

(Previous tests were conducted at 120°F., which has important bearing upon soil removal.)

Detergents. The detergents used in these tests were the same as for Parts I and II:

1. Built nonionic
2. Built alkylaryl sulfonate
3. Pure soap
4. Loralkyl sulfate

Ratio of Load Weight to Solution. Part I of this series indicated that, depending upon the wash machine used, there were considerable differences in the ratio of the weight of fabric in grams, per liter of wash solution. The Launderometer was operated at an initial ratio of 37, with one swatch 6″ x 5″ in size, which was reduced in each 10-minute wash by 1½″ in the 6″ direction of the swatch. The Terg-O-Tometer in these tests was operated with 500 ml. of solution at a load level of either 30 or 60 g. per liter.

Replicate Tests. The Launderometer tests were the averages of five individual duplicate tests. The Terg-O-Tometer tests consisted of five runs, and the 30 g. per liter load contained five replicate samples each while the 60 g. per liter load contained 10 swatches.

Experimental Data and Discussion

Examples of the Launderometer values obtained are shown in Table I. Shown are the individual averages for each of the four 10-minute wash periods as well as the average washing value for each of the five individual four, 10-minute washes.

Representative Terg-O-Tometer values are shown in Table II for the same built nonionic product.

TABLE I

Representative Launderometer Values
Soil Removal Data
Built Nonionic

Test	Wash Period, Minutes				Wash Average
	10	20	30	40	
1	50	54	58	72	58.5
2	50	52	63	69	58.5
3	47	47	56	70	55.0
4	43	46	55	65	52.0
5	62	50	57	75	61.0
Period average	50	50	58	70	57.0

TABLE II

Representative Terg-O-Tometer Values
Soil Removal Data
Built Nonionic

5-Minute Wash			
30 g./l. Load		60 g./l. Load	
60	58	60	56
62	59	60	57
59	57	57	58
60	62	52	70
59	63	61	65
$\bar{x} = 60$	$\bar{x} = 60$		$\bar{x} = 60$

Table III shows a comparison of the soil removal values for each of the four detergents using either machine. For the Launderometer the values are shown either as the four-wash average $\frac{2A+2B+2C+2D}{8}$ or the average values for the third or 30-minute wash period. The third wash period was chosen for comparison purposes because the values closely approach the averages obtained for the four-wash method.

For the Terg-O-Tometer the 30 and 60 g. per liter load ratios are shown for the 5-minute and the 3-minute wash intervals.

It is pertinent that the coefficient of variation, v, is smaller in the Launderometer test for the 30-minute wash average than for the 4-wash average, which might be expected. It furthermore is apparent that, with certain exceptions, the Terg-O-Tometer test values exhibit a lower standard deviation and coefficient of variation than that for the Launderometer test.

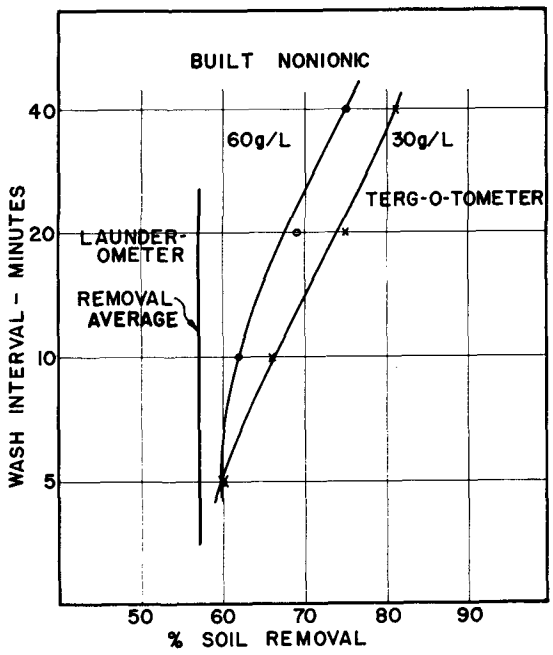

FIG. 1. Comparison of wash methods—nonionic detergent.

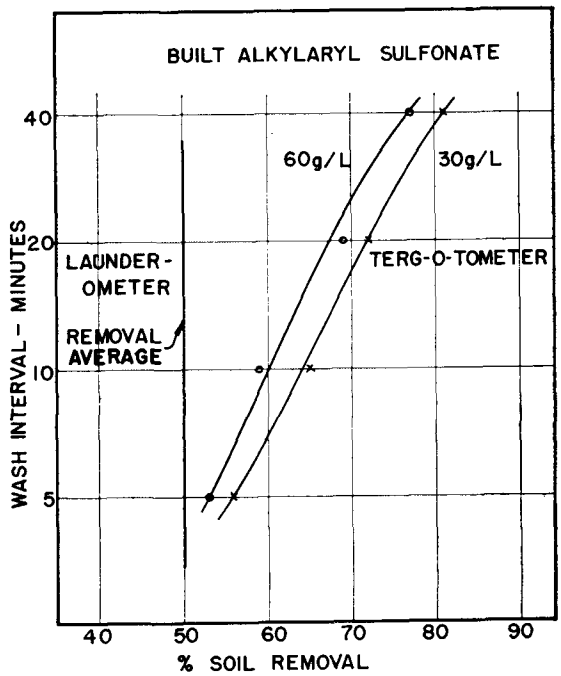

FIG. 2. Comparison of wash methods—built alkylaryl sulfonate.

The effect of wash load in the Terg-O-Tometer is shown by the coefficient of variation. The 30 g./l. load with but one exception is lower than the 60 g./l. load.

This method for evaluation should give essentially the same ratings for the detergents as the previous evaluation data in Parts I and II, where the twice standard error method was used, since both have a 95% confidence limit. Further the present data were newly determined values.

Launderometer. Previous data gave an order to the four detergents as follows:

1st choice—3
2nd choice—1, 2
3rd choice—4

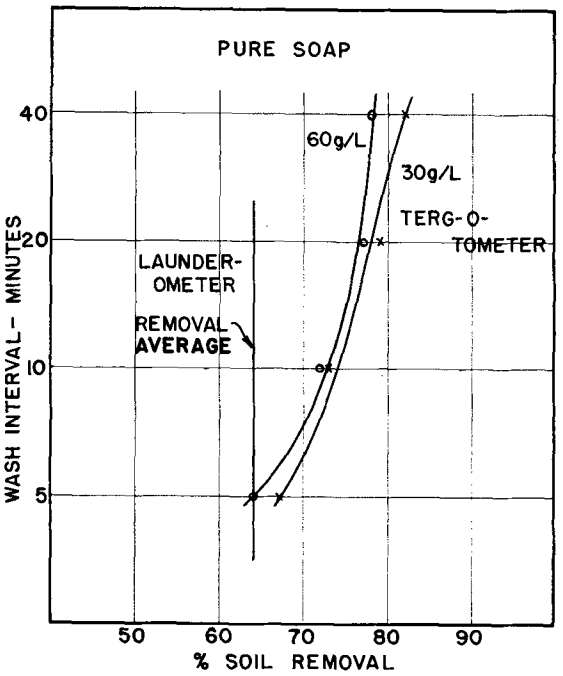

FIG. 3. Comparison of wash methods—pure soap.

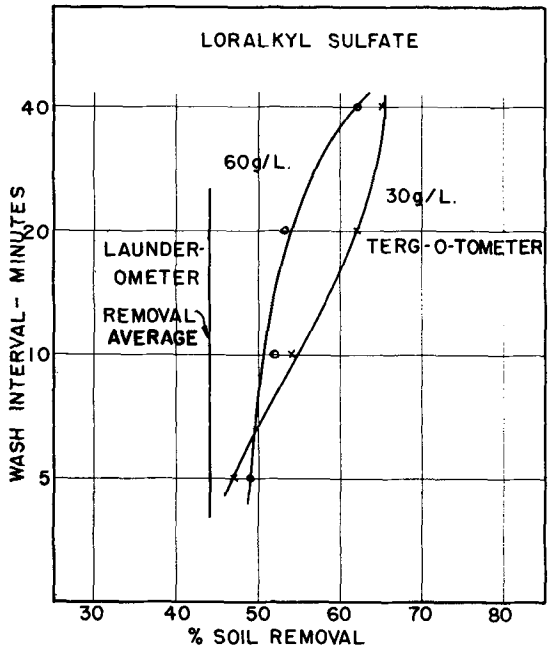

FIG. 4. Comparison of wash methods—loralkyl sulfate.

Figures 1 through 4 show a comparison of the 30- and 60-g. load of fabric per liter of solution for the Terg-O-Tometer test as compared with the four-wash Launderometer average. These data are plotted on log-log paper and indicate that for the four detergents a wash time of about 3 to 4 minutes might provide exactly the same soil removal values for the single wash as for the four-wash average by the Launderometer method. Tests were therefore made at the 3-minute wash period followed by a 2-minute rinse (rather than a 5-minute rinse used for the longer washing periods).

Reference to Table IV shows that the present data give a slightly different order, detergent No. 2 dropping to third choice. The change in rating is slight,

TABLE III

Statistical Data
Five Replicate Tests

Launderometer Tests		$\bar{x}$	σ	v
Detergent	Wash Period			
Built nonionic	30-minute av.	58	2.79	4.8
	4-wash av.	57	3.16	5.5
Built alkylaryl sulfonate	30-minute av.	50	3.10	6.2
	4-wash av.	50	3.44	6.9
Pure soap	30-minute av.	66	3.76	5.7
	4-wash av.	64	4.26	6.7
Loralkyl sodium sulfate	30-minute av.	47	3.82	8.1
	4-wash av.	44	5.54	12.5

Terg-O-Tometer Tests					
Detergent	Wash Period	Load (g./l.)	$\bar{x}$	σ	v
Built nonionic	5	30	60	0.45	0.8
	5	60	60	4.77	7.9
	3	30	59	1.41	2.4
Built alkylaryl sulfonate	5	30	58	1.90	3.3
	5	60	53	2.89	5.5
	3	30	56	1.61	2.9
Pure soap	5	30	61	5.30	8.7
	5	60	60	2.44	4.1
	3	30	55	1.09	2.0
Loralkyl sodium sulfate	5	30	49	1.41	2.9
	5	60	49	2.22	4.5
	3	30	44	0.45	1.0

but significant. Naturally there are 5 chances in 100 for variation, and this may be operative in either this test or the one shown in the previous papers. A further factor which could cause variation is that the present tests were conducted at 140°F., instead of 120°F.

Table IV shows a comparison of detergents as obtained by the two wash methods and for each of the variations within the methods. In this case the mean

TABLE IV

Comparison of Detergents
$\bar{x} \pm a\sigma$ where $a = 1.388$, with 95% Confidence Limit

Launderometer			Terg-O-Tometer		
			3-Min. Wash	5-Min. Wash	5-Min. Wash
Rating	30-Min. Wash	40-Min. Av. Wash	30 g./l. Load		60 g./l. Load
1st	3	3	1	1, 3	3, 1
2nd	1	1	2, 3	2	2, 4
3rd	2, 4	2, 4	4	4	

1—Built nonionic.
2—Built alkylaryl sulfonate.
3—Pure soap.
4—Loralkyl sodium sulfate.

values were compared by determining whether there were actual differences between them by applying a 95% confidence limit (5). For five replicate samples the mean value in 95 cases out of 100 has a range of $\bar{x} \pm 1.388\,\sigma$.

It might be reasoned from observation of the comparison between the 30-minute wash and the 40-minute wash average that a single 30-minute wash might be operated. However this is not at all the same mechanism that was used in these tests. The mean value of all four washes will average out variables inherent to the multiple suds method.

With constant mechanical action there may be considerable differences in the rate at which soil is removed using different detergents. Figure 5 shows the detergency curves obtained with the four detergents in the Launderometer test. They show that the slopes

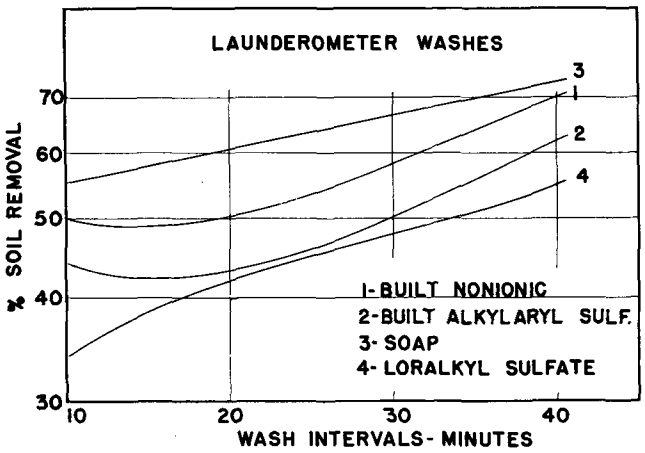

FIG. 5. Soil removal curves for the Launderometer method.

of the initial 10-minute wash periods vary considerably and that the slopes flatten off after that period. The average slopes of the two built products are essentially equal to those for the other two products but the rates of removal at the end of the fourth wash period are about double those of the other detergents.

Terg-O-Tometer. A different arrangement of detergents is apparent at the 30 g./l. load level, when comparing the 3- and 5-minute wash intervals. This difference may be a function of the wash-and-rinse time since there is sufficient difference in the soil removal values to indicate differences in behavior.

It might be thought that continued washing might ultimately produce fabrics of the same level of reflectance, but that this does not occur in the Launderometer four 10-minute wash periods is very evident. It is pertinent that four 10-minute washes in the Launderometer yielded lower soil removal values than a single 40-minute wash in the Terg-O-Tometer at the 30 g./l. load level.

Reference to Figures 1 through 4 indicates that for the Terg-O-Tometer the slope of the soil removal curve is greater for the 30 g./l. load than when 60 g./l. load is used. This difference may be attributed to reduction in mechanical action, probably as a result of overloading.

Experience over the years has shown the Launderometer 40-minute wash test as a reliable indicator of commercial application. Because the 30 g./l. load in the Terg-O-Tometer for a 3- or 5-minute wash period most closely approached the Launderometer results, the 5-minute wash was chosen for further investigation even though the arrangement of the four detergents in relative order of effectiveness was not good. An extended series of tests was made in which the same soil was used for either wash test method, and detergent samples of different chemical structure and soil removal characteristics were chosen for investigation. In each of these washes loralkyl sodium sulfate was used as a reference standard.

Comparison of the values of Table V is made arbitrarily, using the largest individual standard deviation for the detergents by each of the test methods. For the Launderometer this was 5.54 for loralkyl sulfate and for the Terg-O-Tometer 5.30 for soap. These standard deviation values were multiplied by the "a" factor of 1.38 (p. 43, Table II of the ASTM Manual on Quality Control of Materials) for 5 observations, each at a 95% confidence limit, giving the values shown in Table V.

This shows that all the comparative values for these two tests fall within the 95% confidence limit.

These data, based upon a relatively extended series of samples, show rather better correlation than was obtained with the four-detergent series and there was good correlation between the two wash test methods.

TABLE V

Comparison of Test Results With a Variety of Samples
50 ppm. Water
95% Confidence Limits
$a\sigma$ Value for Launderometer—7.7
$a\sigma$ Value for Terg-O-Tometer—7.4

Sample	Soil Removal, Launder-ometer	Soil Removal, Terg-O-Tometer [b]
4756	30.5	34
4765	17	15
Standard [a]	41	39
4769	50.5	50
4771	19.5	12
Standard	51	49
4787	46.5	45
4788	25	27
4789	33	36
Standard	47	53
4790	25.5	31
4791	29	32
4792	42	41
Standard	45.5	46
4820	36.5	43.5
4810	8.5	13
4817	56	58.5
Standard	43	47

[a] Loralkyl sodium sulfate.
[b] 30 g./l. load, 5-min. wash.

Conclusions

It was demonstrated that the average soil removal values for the four 10-minute wash Launderometer method could probably be essentially duplicated by a single 3-minute Terg-O-Tometer wash and one 2-minute rinse. The 3-minute Terg-O-Tometer method did not result in the same comparative rating of four detergents, but a series of tests with several other detergents using a 5-minute wash and a 5-minute rinse (using for comparison purposes a standard detergent) resulted in quite effective ratings. Of 13 detergents tested over a period of time, the two test methods provided soil removal values identical within a 95% confidence limit.

These data indicate that a satisfactory technique can be developed using the Terg-O-Tometer, which would provide soil removal values and ratings of detergents closely paralleling the results obtained by the Launderometer multiple-wash method.

Acknowledgment

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