Performance Characteristics of Sucrose Ester Detergents

ANTHONY M. SCHWARTZ and CHARLES A. RADER, Harris Research Laboratories, Inc., Washington, D.C.

Abstract

Standard heavy duty household laundering formulations were prepared from a series of well-characterized sucrose ester surfactants. The detergency and redeposition performance of these formulations was measured by the soil accumulation method, using for comparison standard formulations based on sulfated and sulfonated surfactants. Also measured were the lime soap dispersing power, and the tendency to leave residual adsorbed deposits on the fabric. The sucrose ester series as a whole performed at least as well in detergency as the standard anionics, and retained their effectiveness at lower concentrations. They showed about the same, or slightly less, tendency than the anionic controls to build up organic residues on the fabric, both series being much superior to soap in this respect. In redeposition performance and lime soap dispersion the sucrose esters were outstandingly better than any of the standard anionic controls. Within the sucrose ester series, the C_{18} esters gave generally better performance than the shorter chain esters, and the saturated esters tended to be better than the unsaturated.

Introduction

Although the surface active fatty acid esters of sucrose have received considerable attention since their preparation on a practical scale was first described, relatively few studies of their detersive properties have appeared in the literature (1). Their recent availability in commercial quantities, together with their high biodegradability and physiological inertness, has stimulated renewed interest in their practical detergent possibilities. The purpose of the present work was to explore the detersive power and other practical performance characteristics of representative commercially available sucrose ester surfactants when made up into a typical heavy duty household laundering detergent. The following performance characteristics were determined: detergency on carbon soil cloth, detergency on vacuum cleaner soil by the soil accumulation method, foam in the washing machine, lime soap dispersion, and deposition of insoluble residue on the fabric over a prolonged series of washing cycles.

Materials

I. Sucrose Esters

Five different sucrose esters were used, furnished by three different manufacturers identified herein as Companies A, I, and J, as follows: A tallow ester from A; a stearate ester from A; a tall oil fatty acid ester from A; a laurate ester from I; and a coco fatty acid ester from J. The first four materials were solids of high active ingredient content. The coco product was an aqueous paste. These substances were analyzed as follows, using the standard methods recommended by the Sugar Research Foundation, Inc., for sucrose esters.

The percentage moisture and volatile matter was determined first. The dry residue from this determination was then extracted with ethanol to obtain the percentage of alcohol-soluble organic matter. This percentage (Row 2 in Table I) was used as the "active ingredient" (abbreviated a.i.) content in making up the final detergent formulations. The nonvolatile, alcohol-insoluble material consisted largely of sugar and inorganic salt. The free fatty acid and neutralized fatty acid (soap) contents of the original samples were then determined, and the percent sucrose ester was calculated by subtracting these values from the alcohol-soluble value. Samples of the original material were saponified, the fatty acids were isolated, and their molecular weights were determined by titration with alkali. During this procedure care was taken to make the yield of fatty acid as nearly quantitative as possible, and the actual yield was noted. Saponification equivalent determinations were then run on the alcohol-soluble material. From these saponification equivalents and the molecular weights of the fatty acids, the percentages of monoester and diester were calculated. As an independent check, the percentages of monoester and diester were also calculated from the yields of fatty acid obtained in the saponification step, assuming these yields to be quantitative.

The analytical data are shown in Table I. For purposes of discussion the tallow, tall oil and coco products can be regarded as essentially monoesters. The stearate and laurate, as mixtures of roughly equal parts monoester and diester.

II. Comparison Surfactants

For comparison with the sucrose esters, in formulated form, the following surfactants were used, all

TABLE I

Analysis of Sucrose Esters

	Coco ester (J)	Tallow ester (A)	Tall oil ester (A)	Stearate ester (A)	Laurate Ester (I)
1. % Alcohol soluble	47.1	97.6	93.3	92.5	83.3
2. % Moisture and volatiles	49.7	.6	.3	.3	2.0
3. % Non-volatile and insoluble in alcohol (100-line 1-line 2)	3.2	1.8	6.4	7.2	14.7
4. % Free fatty acid	1.2	.5	.5	.3	1.5
5. % Soap	1.8	.6	5.5	.4	3.4
6. % Ester (line 1-line 4-line 5)	44.1	96.5	87.3	91.8	78.4
7. % Total fatty acid isolated	20.3	42.3	46.2	52.5	42.7
8. % Fatty acid combined as ester (line 7-line 4-acid equivalent of line 5)	17.5	41.2	40.6	51.8	38.1
9. M.W. of fatty acid	227	278	276	276	196
10. % Fatty acid in ester (100 × line 8/line 6)	39.6	42.7	46.5	56.5	48.6
11. [a] Mono ester/diester ratio	100/0	100/0	98/2	43/57	41/59
12. Found saponification equivalent of alcohol soluble material (line 1), corrected for free acid (line 4) = sap. equiv. of ester (line 6)	518	598	569	492	408
13. [b] Mono ester/diester ratio	85/15	98/2	86/14	45/55	45/55

[a] Calculated using the found % fatty acid (line 10) and the theoretical values for % fatty acid in sucrose mono ester and diester (based on the experimentally determined MW of the fatty acid).

[b] Calculated using the found saponification equivalent (line 12) and the theoretical values of saponification equivalents for sucrose mono ester and diester (based on the experimentally determined MW of the fatty acid).

of which were obtained from commercial sources: 1) sodium α-sulfo myristate; 2) a mixed fatty alcohol sulfate sodium salt, prepared by mixing 80 parts of lauryl sulfate with 20 parts of tallow alcohol sulfate; 3) a low-salt-content branched chain alkyl benzene sulfonate (ABS); 4) two different samples of straight chain alkyl benzene sulfonate (LAS) from different manufacturers, designated LAS-B and LAS-C. These materials were checked for active ingredient content; 5) flaked low titer and high titer soaps used as received in the fabric deposition and lime soap dispersion tests; 6) a nonylphenol-10 ethylene oxide nonionic surfactant (nominal 100% a.i.), used in the lime soap dispersion tests.

III. Formulations

Formulations were made up from the surfactants as follows: Each 100 parts of total formulation contained 40 parts sodium tripolyphosphate (NaTPP), 1 part ("as is" basis) carboxymethyl-cellulose (NaCMC), and 6 parts (dry basis) sodium silicate. The NaCMC was Hercules Powder Co. Type 7LT. The sodium silicate was Philadelphia Quartz Co. brand N. The remaining 53 parts were composed of surfactant (a.i. basis) and sodium sulfate, the proportions varying as shown in the tables from 5 surfactants—48 Na_2SO_4 to 25 surfactant—28 Na_2SO_4. A representative sample of sucrose ester formulation was analyzed for NaCMC content by a proposed ASTM method (8) and found to contain 0.82%.

Two formulations which were not made up this way but were procured in the already formulated state are identified as "Committee LAS" and "Commercial Detergent." "Committee LAS" was obtained from the Research Committee of the American Association of Textile Chemists and Colorists, Washington Section. It contained 19% LAS, by analysis; together with NaTPP, NaCMC, and silicate in conventional proportions. The NaCMC content, as determined by the ASTM Method, was 0.59%. The commercial detergent was a leading brand of the high foaming type based on anionic surfactants. It was purchased at retail, analyzed by titration with cetyltrimethylammonium bromide using bromophenol blue indicator, and found to contain 18.8% anionic a.i. (assumed to be dodecyl benzene sulfonate). The NaCMC content, as determined by the ASTM Method, was 0.33%.

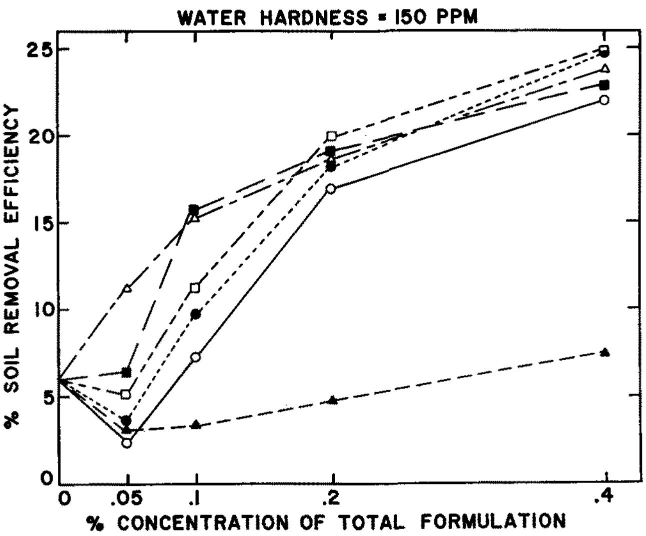

FIG. 1. Carbon soil detergency in 150 ppm hard water. Code for Figures 1 and 2: open triangles, sucrose tallowate; solid triangles, builders only; open circles, sucrose laurate; solid circles, fatty alcohol sulfate mixture; open squares, commercial detergent, purchased; solid squares–Committee LAS.

Experimental

Detergency by the Carbon Soil Method

Carbon soil detergency tests were carried out on U.S. Testing Co. cotton soil cloth, using both 150 ppm and 300 ppm hard water. The Terg-O-Tometer was used as the washing device. The swatches were given one × 10 minute wash at 140F (60C). In the Terg-O-Tometer, the degree of rotation was 380° and the speed of rotation was 95 per minute. They were then rinsed thoroughly in two changes of tap water at 140F, squeezed, dried, and read on the reflectometer. Each pot of the Terg-O-Tometer contained one liter of wash liquor and ten 4 in. × 6 in. soil cloth swatches. No redeposition data was taken, i.e., no white redeposition swatches were included. Washings were carried out at concentrations of zero, 0.05%, 0.1%, 0.2%, and 0.4% total washing formulation. The results are expressed in Figures 1 and 2 as curves showing percent soil removal efficiency (%SRE) vs. concentration. The %SRE is calculated according to the formula;

$$\%SRE = \frac{R_L - R_S}{R_O - R_S} \times 100$$

where

R_L = reflectance of the laundered swatches
R_S = reflectance of the soiled swatches
R_O = reflectance of original unsoiled fabric

In these runs R_O was in the neighborhood of 90 (90% whiteness referred to standard white plaque) and R_S in the neighborhood of 25.

The formulations with sucrose tallowate, sucrose laurate and fatty alcohol sulfate were made up to contain 25% a.i. and 28% sodium sulfate. The "builders-only" formulation contained 53% sodium sulfate. Since these formulations all contained 40% NaTPP, it can be estimated (2) that there was sufficient NaTPP to overcome the water hardness of 150 ppm at washing concentrations of 0.1%, and higher. At the water hardness of 300 ppm there was sufficient NaTPP to completely soften the water at washing concentrations of 0.2%, and higher.

Detergency by the Soil Accumulation Method

For more realistic comparisons of detergency among

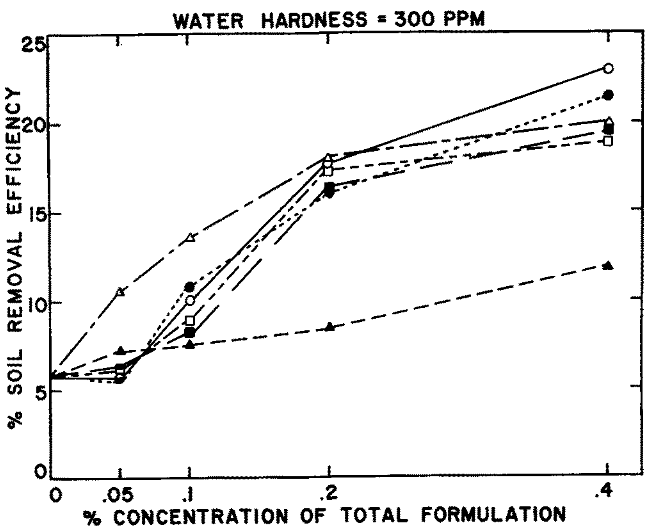

FIG. 2. Carbon soil detergency in 300 ppm hard water.

the various formulations the soil accumulation method described by Schwartz and Berch (3) was used. Since the quantity of soil applied to the swatches in this method is randomized rather than standardized, the test is a head-to-head comparison in the Terg-O-Tometer of the detergents in question against one or more standard detergents, samples of which must be included in each run. It does not measure the amount of soil removed from dirty swatches in any one laundering treatment, but the amount of soil retained by the swatches after a series of soiling and laundering cycles. Included in each of the laundering cycles, but not in the soiling cycles, are a set of white "control" or "redeposition" swatches. These are never deliberately soiled but pick up soil only from the laundering baths as these baths become fouled through washing the dirty swatches. This type of test is regarded as a good predicter of performance under practical home laundering conditions (4,5).

All tests were run in 150 ppm synthetic hard water (70–to–30 Ca/Mg ratio). A total of 5 washing-soiling cycles was used, each washing being run at 60C for 10 minutes. The fabric was Testfabrics No. 400M mercerized cotton printcloth. The Terg-O-Tometer baths were 1 liter, in which 10 swatches 4 in. × 6 in. were washed, 7 soiled and 3 redeposition or control. A single large lot of vacuum cleaner dirt was used. Soil slurry was prepared by extracting 1 kg of the dirt with a total of 6.5 l tap water. This soil slurry was used for all soiling cycles, at a dosage of one liter slurry per 84 swatches. The concentration of total formulation in all runs was 0.4%.

The results are presented as actual Reflectometer reflectance readings at the end of the fifth washing cycle. The Reflectometer was adjusted to eliminate the effect of optical brighteners in those comparison detergents that contained them.

Foaming in Washing Machine

Hand towels were soiled in the vacuum cleaner soil slurry, using the same proportions of soil to fabric as in the preparation of swatches for the soil accumulation detergency tests. Six pound loads of these soiled towels were laundered in a top loading washing machine, adding the load one pound at a time and observing the foam-killing effect of each increment.

Commercial detergent, a 20% a.i. sucrose tallow ester formulation, and a 20% a.i. sucrose laurate ester formulation were compared. The amount of 150 g of total detergent formulation was dissolved in approximately 12 gallons of water (the "high" water level) in the machine. Exactly the same water level was used in each of the three runs. Thus the approximate concentration of detergent formulation in the wash liquor was 0.33%. In each run the temperature of the wash liquor was 126 ± 2F.

The height of the foam was measured initially after the detergent dissolved, and then measured after each addition of 1 lb of soiled cotton huck towels up to a final load of 6 lb of soiled towels. The foam was judged for height, surface coverage, and appearance.

The towels were soiled as follows: 4500 g of vacuum cleaner soil was extracted with a total of 24 liter of water. This soil slurry was then diluted to 40 liter with water and 18 lb of towels were soiled together in this soil-containing liquor. The towels were then squeezed through nip rolls to about 100% wet pick-up, tumble dried, randomized and sorted into 18 piles of 1 lb each to be used in the 3 suds observation tests (6–1 lb piles of towels for each test).

Lime Soap Dispersion

Lime soap dispersing power is the ability of a surfactant to prevent or inhibit the formation of visible, filterable clots of lime soap when dissolved calcium is added to a solution containing ordinary soap and the surfactant in question. This property, of sucrose tallowate and of two comparison surfactants, was measured by two different procedures. The first procedure described by Harris (6), uses a soap concentration of about 1% and the results are expressed as centigrams of agent required to disperse 45.5 mg of calcium oleate at the stipulated dilution. The lower the number the greater the lime soap dispersing power.

The second procedure by Knowles, et al. (7) uses a soap concentration of 0.1% or less, which is more typical of actual working conditions. This procedure was also extended to test the dispersing power at a soap concentration of 0.02%, typical of the severe conditions of rinsing in hard water. In this test the lime soap dispersing value of a surfactant is reported as the lowest percentage of surfactant in a soap-surfactant mixture which prevents clotting of lime soap at a water hardness of 360 ppm (as $CaCO_3$) where calcium and magnesium are present in the ratio of 3 to 1.

For both the Harris test and the test of Knowles et al., (also referred to as the Nessler tube test) the same two comparison surfactants were used. One was the low-salt-content ABS, a relatively poor lime soap disperser. The other was the nonyl phenol-10 ethylene oxide nonionic, considered a good lime soap disperser.

Deposition of Residues on Fabric

Two series of launderings were carried out to compare the performance of the sucrose esters with that of commercial detergent and soap in regard to deposition of residues on the laundered fabrics.

In the first series the launderings were done at a water hardness of 300 ppm and a temperature of 60C (140F). The rinses were in tap water (approximately 100 ppm hardness).

In the second series the laundering temperature was 50C (122F). 300 ppm hardness water (prepared from distilled water, using $CaCl_2$ and $MgCl_2$ in 70 to 30 molar ratio) was used for both laundering and rinsing.

Each series was run for a total of 30 cycles. Clean (not soiled) 80 × 80 cotton printcloth was used.

Ten 4 × 6 in. cloth swatches were laundered in one liter of washing bath containing 0.4% total formulation. The swatches were oven-dried between launderings.

The laundering formulations used were: *1st Series*

A—Commercial detergent

B—Crutched sucrose tallowate formulation, 20% a.i., 40% NaTPP, 1% NaCMC, 6% Na silicate, 33% Na_2SO_4

C—20% high titer soap, 40% NaTPP, 6% Na silicate, 1% NaCMC, 33% Na_2SO_4

2nd Series

A, B,—Same as 1st series

C—20% low titer soap, 40% NaTPP, 6% Na silicate, 1% NaCMC, 33% Na_2SO_4

After 30 launderings, the fabrics were checked for weight change and for reflectance. The weight change is simply a check on fiber loss or excessive deposition. The deposition was measured quantitatively by alcohol extraction. The reflectance value measures any tendency the detergent may have to impart yellowness to the fabric.

Results and Discussion

Carbon Soil Detergency

Although detergency tests on carbon soil cloth are not generally considered reliable indexes of practical performance, the curves of detergency vs. concentration furnish at least one very useful item of information. They indicate the concentration range at which a detergent begins to exert a significant cleaning effect. In 150 ppm water (Fig. 1) the tallow ester formulation shows significant washing action at 0.05% and correspondingly increased effectiveness at the higher concentrations. The other formulations do not exert significant detersive effect below 0.1 to 0.2%. Above 0.2% there is little if any difference among the formulations with respect to the slope of their detergency—concentration curves. In 300 ppm water (Fig. 2) the effectiveness of tallow ester at low concentrations is even more marked. The other formulations resemble one another quite closely, although there may be some tendency for the fatty derivatives (sucrose laurate and fatty alcohol sulfate) to reach the detergency plateau before the petrochemical derivatives (commercial detergent and Committee LAS).

Soil Accumulation Detergency

Results of the soil accumulation tests are presented in Table II, each group showing the data from one series of comparisons. The relative effectiveness of any two detergents is judged by comparing the extremes of the confidence limits. If these overlap the detergents are judged to be equal. If they fail to overlap, the detergent giving the highest reflectance value is judged superior. Committee LAS was used as a standard comparison detergent in all the series, and at least one other comparison detergent was used in each series to allow inter-series comparisons to be made.

The rankings of the detergents in Series 1 can be summarized as follows: In soil removal fatty alcohol sulfate $\geqq$ tallow ester $>$ coco ester $>$ Committee LAS. In redeposition tallow ester $>$ coco ester $>$ fatty alcohol sulfate $>$ Committee LAS. Series 2 ties in two other sucrose esters and one more comparison detergent at 25% a.i. content. The data show that there is no significant difference among the four detergents of Series 2 with regard to soil removal. In redeposition the ranking is tall oil ester $\geqq$ laurate ester $>$ sulfomyristate $>$ Committee LAS. To complete the group of sucrose esters, the stearate ester was tested at 25% a.i. content against Committee LAS (Rows 1 and 3 in Series 3) and against the tallow ester at 10% a.i. content (Rows 4 and 5 in Series 3). The rankings in soil removal are stearate ester = tallow ester $>$ Committee LAS: in redeposition stearate ester $>$ tallow ester $>$ Committee LAS.

It is evident from the above data that the sucrose esters as a group are comparable in soil removal to the two fat-derived comparison detergents, and are unquestionably superior in redeposition. The relatively poor showing of Committee LAS could be due to its low content of NaCMC, a point which will be considered later. It was now of interest to consider how well these materials performed at lower concentrations. Referring back to Series 1, tallow ester at 25% a.i. was roughly 15 points better in soil removal than Committee LAS. In Series 3, stearate ester at 25% a.i. was about 14 points better than Committee LAS, suggesting that in a head-to-head comparison the stearate and tallow esters would be about equal. The data in Series 3 show that they are about equal in soil removal at 10% a.i., although the stearate is superior in redeposition. Both these sucrose esters are less effective at 10% a.i. than at 25% a.i. but they are at least as effective as Committee LAS. A remarkable result in this series is that the tallow ester was just as effective, both in soil removal and redeposition, at 5% a.i. as at 10% a.i. It is also noteworthy that the fatty alcohol sulfate maintained its effectiveness at 10% a.i. just as well or better than the sucrose esters. As in the group of Series 1, it was superior to tallow ester (more so in Series 3 than in Series 1) in soil removal, but inferior in redeposition.

TABLE II

Soil Accumulation Tests

Detergent	Reflectance at end of 5 cycles 95% confidence level	
	Soil removal	Redeposition
Series 1. Sucrose tallow and coco esters		
Committee LAS	714.3±2.0	765.9±1.4
Fatty alcohol sulfate, 25 a.i.	736.3±2.6	782.1±2.5
Sucrose tallow ester, 25 a.i.	731.2±2.5	798.7± .7
Sucrose coco ester, 25 a.i.	725.1±2.4	790.2± .9
Series 2. Sucrose tall oil and laurate esters		
Committee LAS	685.4±3.1	744.8±1.4
Sodium alpha-sulfo myristate, 25 a.i.	690.0±2.3	761.6±1.6
Sucrose tall oil ester, 25 a.i.	691.6±3.9	771.6±2.5
Sucrose laurate ester, 25 a.i.	689.6±3.5	767.7±1.4
Series 3. Effect of lowered a.i. concentration		
Committee LAS	734.9±3.1	768.1± .7
Fatty alcohol sulfate, 10 a.i.	743.4±2.9	771.0±4.6
Sucrose stearate ester, 25 a.i.	748.4±2.4	788.2±2.0
Sucrose stearate ester, 10 a.i.	737.5±1.6	786.5±1.1
Sucrose tallow ester, 10 a.i.	737.0±2.7	781.2±2.3
Sucrose tallow ester, 5 a.i.	742.8±2.6	780.7± .9
Sucrose tall oil ester, 10 a.i.	731.6±4.4	781.6±2.0
Series 4. Sucrose stearate and tallow esters and various alkyl benzene sulfonates		
Committee LAS	704.6±3.1	770.8±2.9
LAS-C, 20 a.i.	703.2±3.3	787.6±2.0
LAS-B, 20 a.i.	726.2±3.7	793.8±1.9
ABS, 20 a.i.	714.8±4.2	791.8±2.9
Commercial detergent	705.8±2.1	766.4±1.3
Sucrose stearate ester, 20 a.i.	712.0±2.2	803.0±1.7
Sucrose stearate ester, 20 a.i., crutched	710.5±3.3	801.3±2.0
Sucrose tallow ester, 5 a.i.	714.9±3.3	791.6±2.0

Since the mediocre performance of Committee LAS was unexpected, the final series of runs included two LAS samples and one ABS sample formulated to exactly the same a.i. content and NaCMC content as the sucrose stearate ester. The commercial detergent, and a check run of the tallow ester at 5% a.i. were also included. The results are shown in Series 4. The two samples of LAS differed greatly in soil removal, the LAS-B being much superior to any of the other samples in the series. The LAS-C gave about the same soil removal performance as Committee LAS and Commercial Detergent. The ABS, stearate ester at 20% a.i. and tallow ester at 5% a.i. were all about equal to one another in soil removal, and ranked between the LAS-B and Committee LAS. In redeposition the stearate ester was best by a considerable margin. The two LAS samples, ABS, and the tallow ester at 5% a.i. were all quite close to one another in second place. Committee LAS and Commercial Detergent were poorest. The poor showing of Commercial Detergent was doubtless due, at least in part, to its low NaCMC content.

These results may be summarized as follows: the sucrose esters in conventionally formulated form provide outstanding antiredeposition performance; and soil removal at least equal to the favored conventional anionic surfactants. The 18 carbon chain materials give better detergent performance than the shorter chains, and are remarkable for their effectiveness at low a.i. concentrations.

Foam Performance

Results of the foam performance tests are shown in Table III. The laurate resists the foam-killing effects

TABLE III

Foam Behavior of Sucrose Ester Formulations

Wt. of soiled towels added and elapsed time after addition	Commercial detergent			Sucrose tallow ester			Sucrose laurate ester		
	Height of suds, cm	Cover-age	Suds con-sistency	Height of suds, cm	Cover-age	Suds con-sistency	Height of suds, cm	Cover-age	Suds con-sistency
Initial mixing with no fabric	14–15[a]	Full	Lacy	15	0.75	Lacy	15	Full	Lacy
1 lb—1 min	5.5	Full	Creamy	3–3.5	0.75	Creamy	6.5–7	0.9	Creamy
2	5.5	Full	Creamy	3–3.5	0.875	Creamy	6.5–7	0.9	Creamy
2 lb—1 min	4	0.875	Creamy	1	0.9	Creamy	5–6	0.875	Creamy
2	4	0.875	Creamy	1	0.9	Creamy	5–6	0.875	Creamy
3 lb—1 min	4	0.875	Creamy	0.5–1	0.9	Creamy	4	0.875	Creamy
2	3	0.66	Creamy	0.5–1	0.9	Creamy	3	0.75	Creamy
4 lb—1 min	2.5	0.66	Creamy	0.5–1	0.75	Creamy	1.5	0.75	Creamy
2	2.5	0.66	Creamy	0.5–1	0.875	Creamy	1.5	0.75	Creamy
5 lb—1 min	2.5	0.66	Creamy	0.5	0.875	More lacy	0.5	0.625	Creamy
2	1.5	0.5	Creamy	0.5	0.875	More lacy	0.25	0.875	Creamy
6 lb—1 min	1.5	0.5	Creamy	0.5	0.5	More lacy	0.25	0.333	Creamy
2	1.5	0.5	Creamy	0.5	0.5	More lacy	0.25	0.5	Creamy

[a] 15 cm is the distance to top of machine.

of soil somewhat better than the tallow ester, although after 5 lb of soiled towels have been added there is little to choose between them. Neither material was as persistent as Commercial Detergent, although up until 4 lb of towels had been added the laurate ester was as good as Commercial Detergent. It is noteworthy that the sucrose ester formulations contained no foam booster but nevertheless performed much more like a high-foaming type than like a controlled-foaming type.

Lime Soap Dispersion

Data on the lime soap dispersion tests are shown in Tables IV and V. Both tests show the sucrose ester about equal and quite similar to the nonionic, and much superior to the anionic. In the Nessler tube test, this superiority is more accentuated at the higher dilution.

Unfortunately, neither of the tests which were used (nor any of the laboratory tests for lime soap dispersion of which we are aware) can be translated directly into performance under practical conditions. It is a general rule that the lower the concentration of soap and surfactant in a hard water medium the more difficult it becomes to keep the lime soap dispersed. It also becomes more difficult to make a quantitative estimate of the precipitation: therefore the tests are made at surfactant concentrations higher than those encountered in a typical rinse cycle. The actual percentage of sucrose ester that one would have to put into a soap bar or soap powder to prevent hard water scumming is probably higher than the percentages indicated in the tests above. The relative rating of the surfactants, however, is reliable. Sucrose ester has some advantage over the polyethylene oxide nonionics in being easier to incorporate into a solid formulation.

TABLE IV

Lime Soap Dispersion Numbers by Harris Test

Detergent	Dispersion number
Sucrose tallowate	10 to 20
ABS	50
Nonionic	10 (lowest value tested)

TABLE V

Lime Soap Dispersion Values—Nessler Tube Test

	Dispersion Value, in %	
Surfactant	At .1% conc.	At. .02% conc.
Sucrose tallowate	Less than 10	Less than 10
ABS	30	40
Nonionic	Less than 10	Less than 10

TABLE VI

Alcohol Extractable Residue on Laundered Fabrics

	% Residue on fabric	
Laundered in:	1st Series	2nd Series
Commerial detergent	0.28	0.19
Sucrose tallow ester	0.25	0.08
Soap	0.51	2.06

Deposition of Residues

Results of the deposition tests are shown in Table VI. The test for accumulation of residues is fully realistic, and shows the excellence of the sugar esters in comparison with soap. Formulated sugar ester is at least as good as Commercial Detergent and probably somewhat better. Part of the 0.19% residue from Commercial Detergent (Table VI, second series) is probably due to the foam-stabilizing constituent. The difference between the series 1 and series 2 deposition levels of the non-soap surfactants is probably due to the different temperatures of washing and to minor differences in the extraction procedure. It is interesting that soap gives high deposition even though it is formulated with an excess of tripolyphosphate and produces perfectly clear high-foaming wash liquors. The deposition evidently takes place in the rinsing cycle, and is much greater in 300 ppm rinse water than in 100 ppm rinse water. The quantity of free soap in the sucrose ester formulation is sufficiently low, and the dispersing power of the ester itself is sufficiently high, to prevent any significant deposition even under the severe conditions of the series 2 runs.

In neither series was there an significant difference in final reflectance among the three samples, after correcting for the optical brightener effect.

ACKNOWLEDGMENT

We wish to thank the Sugar Research Foundation Inc., under whose sponsorship this work was undertaken, for permission to publish. We also wish to thank Miss Elaine Huey, Miss Grace Richardson and Miss Barbara Besterman for assistance in making the measurements.

REFERENCES

1. L. Osipow, F. D. Snell, D. Marra and W. C. York, Soap Chem. Specialities *32* (8), 57 (1956).
2. P. T. Vitale, J. Ross and A. M. Schwartz, Soap Chem. Specialties *32* (6), 41 (1956).
3. A. M. Schwartz and J. Berch, Soap Chem. Specialties *39* (5), 78 (1963).
4. W. G. Spangler, H. D. Cross III and B. R. Schaafsma, "A Laboratory Method for Testing Laundry Products for Detergency," paper presented at American Oil Chemists Society Meeting, Chicago, October 1964.
5. W. B. Michlik and H. D. Cross III, JAOCS *42,* 177 (1965).
6. Harris, J. C., "Detergency Evaluation and Testing," Interscience, New York, 1954, p. 33.
7. D. C. Knowles, Jr., J. Berch and A. M. Schwartz, JAOCS *29,* 158 (1952).
8. A.S.T.M. Standards, *Part 22,* 429. A.S.T.M., Philadelphia, February 1965.

[Received April 5, 1965—Accepted May 26, 1965]