TABLE II
Fatty Acids in Unremoved Clothes Soil

Carbon atoms	Percentage						
	A			B	C		D[a]
	Total[b]	Free	Lime soap	Total[b]	Total[b]	Free	Total
<C_{12}	1	2	....	1	1	1	6
C_{12}	1	6	....	2	2	6	1
C_{13}	2	3	....	1	1	2	....
C_{14} Myristic	6	24	6	7	9	24	7
C_{14} Unsat.	1	1	....	1	1	1	5
C_{15} Total	11	12	5	10	9	13	10
C_{16} Palmitic	33	30	38	36	33	29	30
C_{16} Branched	2	....	....	2	1	....	....
C_{16} Unsat.	10	7	16	9	11	10	9
C_{17} Total	5	2	3	6	5	4	5
C_{18} Stearic	18	6	26	18	15	5	9
C_{18} Oleic	6	7	6	5	10	5	10
>C_{18}	2	....	1	2	3	....	....

[a] Extracted with ethanol.
[b] Excluding lime soaps.

contains more myristic acid and less stearic acid. According to Rothman (9), there is little difference in the relative distribution of single members in free and esterified skin fat. The presence of large amounts of odd-numbered fatty acids indicates their human source. Odd-numbered normal acids have not been observed in large quantities in other materials besides human sebum (9).

The free fatty acid composition is similar to that reported by Weitkamp *et al.* (11) for hair fat and James and Wheately (2) for sebum except that the organic soil recovered from clothes has a greater amount of myristic, pentadecanoic, and palmitic acids. The amount of oleic is much less than that present in skin and hair fat. This agrees with the observation of Walter (10) that less unsaturated fatty acids are present in built-up clothes soil than in freshly-adsorbed soil. The low amount of oleic and the absence of linoleic and linolenic acids probably result from the oxidation of these compounds to polymers and other oxidation products.

Summary

Organic soil that had gradually accumulated on cotton garments and was unremovable by normal washing procedures was analyzed for free and combined fatty acids by gas-liquid chromatography.

The fatty acid composition of this material was similar to sebum and hair fat and was remarkably uniform although from several different sources and geographical locations. The predominant fatty acids were C_{15}, C_{16}, and C_{18} straight-chain acids. More than 30% of the total fatty acid was palmitic acid. The amount of oleic acid was considerably less than is reported for hair and skin fat. No linoleic acid or linolenic acid was detected. The small amount of unsaturated acids is probably the result of their oxidation to polymers and other oxidation products. The amount of free fatty acids was very small because they were converted to insoluble heavy metal soaps. Most of the combined fatty acids were present as esters, *i.e.*, triglycerides.

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Analysis of Surfactant Mixtures

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ALTHOUGH MANY METHODS have been presented for analysis of surface-active agents, none have actually been based on analysis of built detergent compositions containing surfactants which had been previously characterized. Heretofore most methods have been concerned with a certain phase of surfactant analysis. It is the purpose of this paper to show how surfactants or actives may be isolated from detergent compositions and then how the actives are separated and characterized as nonionics, sulfonates, sulfates, and hydrotropes.

Surfactant Characterization. The first step in this study was to characterize the surfactants which would be used for preparing detergent compositions for subsequent analysis. The surfactants used were commercial products, and their characterization was carried out as follows.

Water content was determined by ASTM D1568-58T (1). a) Xylene distillation method was run according to Sections 9 to 12. b) Karl Fischer method was run according to Sections 13 to 18 for samples with less than 1.0% water.

Alcohol insolubles, sodium chloride, neutral oil, active content, combining weight, SO_3, and cationic titration were determined by ASTM D1681-59T (2).

Reagent grade boiled isopropyl alcohol (Merck) cut to 95% by volume with distilled water was used in place of ethanol. Actives, especially tallow alcohol sulfate, had better solubility in 95% isopropyl alcohol than in 95% ethanol.

Determination of alcohol-insolubles consisted of digesting the surfactant sample [1] in 95% isopropyl alcohol, decanting the alcohol-soluble through an asbestos filter, dissolving the alcohol-insolubles in a minimum of water, precipitating salts from solution by addition of 95% isopropyl alcohol, washing the precipitate on the filter with 95% isopropyl alcohol, and drying and weighing the alcohol-insoluble residue.

The filtrate containing the alcohol-solubles was heated to near dryness, dissolved in 50% isopropyl alcohol (boiled), and then extracted with petroleum ether to remove neutral oils. The petroleum ether was boiled off, and the oil residue was heated for 1 hr. at 105°C.

An aliquot of the active solution at this point was analyzed for sodium chloride by potentiometric titration with silver nitrate. Another aliquot in a tared platinum crucible was evaporated to near dryness, and the active residue was heated to constant weight at 60°C. in a vacuum oven. Following this, the active was ashed while being treated with concentrated sulfuric acid, and the ash was then heated to constant weight in a muffle furnace. Combining weight and SO_3 contents of the active were calculated after correcting for sodium chloride prior to the ashing step and correcting for sodium chloride converted to sodium sulfate after ashing.

Cationic titrations of the samples were made with CTAB (cetyl trimethyl ammonium bromide) previously standardized with pure sodium n-dodecylbenzene sulfonate.

The columnar ion exchange method described by Ginn and Church (3) was used to determine the nonionic active content of nonionic surfactants.

Table I lists data for surfactant characterization from analyses run in duplicate. The dodecylbenzene sulfonic acid had SO_3 contents in perfect agreement by cationic titration and ashing methods. Sodium xylene sulfonate combining weights of 208 and 210 were equal to the theory of 208.

The SO_3 contents by cationic titration and ashing of active for the tallow alcohol sulfate were in fair agreement. Poor agreement was found however for the sulfated nonionic. It was believed that the difficulty may have been caused at least in part by incomplete purification of the active. There may have been unsulfated nonionic left in the sample prior to ashing. The surfactant manufacturer recommended carbon tetrachloride for extraction of unsulfated nonionic. This improved results some, but still fair differences remained. It is possible that traces of inorganic salts in the purified actives may have given the higher SO_3 values after ashing.

The active contents of the nonionic surfactants were based on the ion exchange analysis.

[1] The sulfated surfactant solutions were maintained just slightly alkaline with phenolphthalein as the indicator. This was done throughout their analysis to minimize hydrolysis.

TABLE II

Builder Stock Solution

350.0 g.	Sodium tripolyphosphate
125.0 g.	Sodium silicate, 2 SiO_2/Na_2O, 44% solution
72.5 g.	Sodium sulfate (anhydrous)
10.0 g.	Soda ash (anhydrous)
7.5 g.	Sodium carboxymethyl cellulose, 65% active
2035.0 g.	Water

Volatile at 105°C. is 80.1%.

Detergent Compositions. Three built detergent compositions were prepared using the surfactants previously characterized. The builders, typical of those used in many powder type detergents, are listed in Table II as the formulation used in preparing their solutions. This builder stock solution was mixed with the surfactants in the proportions shown in Table III. The total solution batch size was diluted to about 1,000 ml. for Detergents A and B and to 2,000 ml. for Detergent C. Following this Detergents A, B, and C were drum-dried on steam heated rolls. Detergent A contains built dodecylbenzene sulfonate and sodium xylene sulfonate as the hydrotrope. Detergent B is a combination of built dodecylbenzene sulfonate and tallow alcohol sulfate with lauryl isopropanolamide foam stabilizer and sodium xylene sulfonate hydrotrope. Detergent C has a mixture of dodecylbenzene sulfonate and fatty alcohol + EO (ethylene oxide) condensate as the actives. Considerable trouble was experienced during drum-drying Detergent C in that the dried product was very difficult to remove from the rolls.

Detergent D, a liquid composition, was selected as one containing actives typical of shampoo and hand

TABLE I

Surfactant Characterization

Surfactant	Water by xylene distillation	Alcohol-insolubles	NaCl	Neutral oil	Active	Total found	SO_3 by Ashing	SO_3 by Cationic titration [a]	Combining weight by Ashing	Combining weight by Cationic titration [b]
	%	%	%	%	%	%	%	%	g.	g.
Dodecylbenzene sulfonic acid	4.4	7.8	0.0	0.8	86.8	99.8	21.4	21.4	325	327
	4.4	7.8 [d]	0.0	0.8	87.7	100.8	21.4	21.4	328	327
Sodium xylene sulfonate	2.3	2.7	0.0	0.3	93.5	98.8	35.9	.....	208	208
	2.3	2.6	0.0	0.3	93.6	98.8	35.7	.....	210	Theory
Sodium fatty alcohol-EO sulfate	66.4	0.6	1.3	1.5	28.6	98.4	5.48	4.82	430	.....
	66.0	0.7	1.3	1.4 [e]	28.7	98.1	5.47	4.80	432	476
Sodium tallow alcohol sulfate	63.2	2.2	0.9	6.3	26.4	99.0	6.14	5.97	345	354
	62.8	2.3	0.9	6.2	26.8	98.0	6.32	6.06	338	.....
Fatty alcohol + EO	0.5	0.0	0.0	.....	96.2	.....	.....	.....	.....	.....
	0.5 [c]	0.0	0.0	.....	95.7 [f]	.....	.....	.....	.....	.....
Lauryl isopropanolamide	0.2	0.0	0.0	.....	97.4	.....	.....	.....	.....	.....
	0.2 [c]	0.0	0.0	.....	97.1 [f]	.....	.....	.....	.....	.....
Coconyl diethanolamide	1.1	0.0	0.0	.....	86.1	.....	.....	.....	.....	.....
	1.1	0.0	0.0	.....	86.1 [f]	.....	.....	.....	.....	.....

[a] CTAB standardized by pure sodium n-dodecylbenzene sulfonate.

[b] By cationic titration: combining weight $= \frac{\%\text{ active isolated} \times 80}{\%\ SO_3 \text{ by CTAB}}$

[c] By Karl Fischer.

[d] As H_2SO_4. Sample converted to the sodium salt prior to ASTM D1681-59T analysis.

[e] Extracted with carbon tetrachloride.

[f] From effluent through ion exchange resins.

TABLE III

Detergent Composition

Ingredient	Grams in detergent			
	A	B	C	D
Dodecylbenzene sulfonic acid (neutralized with NaOH)	55.8	16.0	8.0	24.9
Sodium xylene sulfonate	4.8	4.8	.....	.....
Sodium tallow alcohol sulfate	.....	56.4	.....	.....
Sodium fatty alcohol-EO sulfate	.....	.....	.....	50.9
Fatty alcohol + EO	.....	.....	15.6	.....
Lauryl isopropanolamide	.....	6.2	.....	.....
Coconyl diethanolamide	.....	.....	.....	8.7
Builder stock solution	433.0	513.0	634.0	.....[a]
% Volatile at 105°C. after drum-drying	1.1	0.8	1.7	67.0[b]

[a] Water to make 150 g. total for D.
[b] On "as is" basis.

dishwashing detergents. It is based on dodecylbenzene sulfonate, fatty alcohol-EO sulfate, and coconyl diethanolamide foam stabilizer.

Analysis of Actives in Detergent Compositions. The plan followed for analysis of actives in these detergent compositions is given in Figure 1.

A. Separation of Actives from Detergents (Step A)

Reagent grade boiled isopropyl alcohol cut to 95% by volume with water was used in place of ethanol according to ASTM D1681-59T to extract the active ingredients from the detergent compositions. A sample size was selected to yield about 4 g. of active.

SCHEMES FOR ACTIVES ANALYSIS

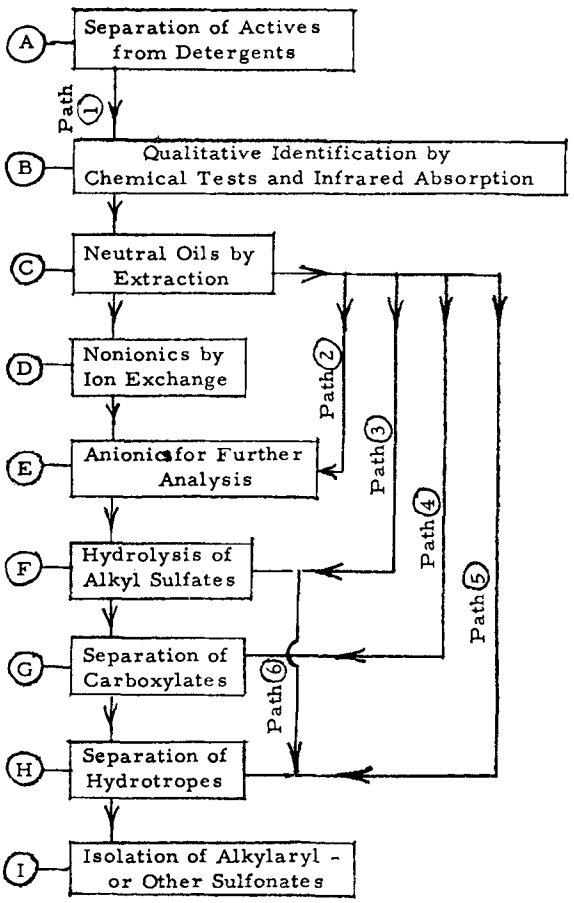

FIG. 1. Path 1 is followed through Step C. Then subsequent steps depend on data from Step B. Step H should be included if sulfates or sulfonates are found in Step B. Path 6 is then followed if carboxylates are absent.

Table IV shows that data by ASTM D1681-59T is in good agreement with theory while those for 24-hr. Soxhlet extractions are quite low for Sample A.

B. Qualitative Identification of Actives (Step B)

Anionics were shown to be present by cationic titration (ASTM D1681-59T).

Alkyl sulfates were identified by reduced cationic titration values for samples that had been refluxed with 2 N sulfuric acid and thereby hydrolyzed.

Nonionics (EO condensates) were present when an acid (pH 4–5) surfactant solution turned blue, and blue precipitate developed after adding cobaltothiocyanate reagent (4).

Nitrogen was identified by the Nessler test. A 4–5-mg. sample was heated in a test tube with one milliliter of concentrated sulfuric acid until fumes developed. After cooling, several drops of 30% hydrogen peroxide were added, and the solution was heated to decolorize organic material. Heating was continued until vapors of sulfuric acid were evident. The peroxide and fuming treatments were continued until the solution was colorless. The solution was cooled in an ice bath, and 10 ml. of 20% sodium hydroxide were added slowly to prevent spattering. Next 2 ml. of Nessler reagent (1.7 g. $HgCl_2$ in 30 ml. of water mixed with 3.5 g. of KI in 10 ml. of water; any precipitate formed was allowed to settle, and the clear solution was decanted) was added. A brown precipitate showed nitrogen to be present. A positive test for nitrogen would be indicated for amides and ammonium salts.

TABLE IV

Isopropyl Alcohol Extraction of Actives

	Found in detergents on anhydrous basis (%)			On "as is" basis (%)
	A	B	C	D
Isopropanol soluble by ASTM D1681-59T	38.3 (38.3)[a] 38.4	28.9 (29.7) 28.9	12.3[b] (15.0) 12.1	32.8 (32.1) 32.7
Soxhlet-extraction	23.8 21.4	29.3 29.4	12.6	

[a] Values in parentheses are theory.
[b] Composition was difficult to drum-dry.

Diethanolamides were identified by Rosen's (5) pyrolysis with sodium chloroacetate.

Infrared absorption spectra (from a Beckman IR-4 spectrophotometer) determined on Nujol mulls of samples between sodium chloride discs revealed the presence of alkylaryl sulfonates, alkyl sulfates, ethylene oxide, and fatty amides.

Qualitative test data listed in Table V are in good agreement with expected results.

TABLE V

Qualitative Identification of Actives

Test	Surfactants from detergent			
	A	B	C	D
Anionic by cationic titration	+	+	+	+
Alkyl sulfate by acid hydrolysis and cationic titration	−	+	−	+
Nonionic by cobaltothiocyanate	−	−	+	−
Nitrogen by Nessler	−	+	−	+
Diethanolamide by pyrolysis with sodium chloroacetate	−	−	−	+
Infrared absorption spectra	AAS	AAS FAM AS	AAS EO	AAS FAM AS EO

AAS—Alkylaryl Sulfonate, FAM—Fatty Amide, AS—Alkyl Sulfate, EO—Ethylene Oxide.

C. Quantitative Analysis of Actives

Neutral oils were extracted with petroleum ether according to ASTM D1681-59T (Step C). The petroleum ether was boiled off, and the residue was heated for 1 hr. at 105°C. After being weighed, the neutral oils were qualitatively tested for nitrogen by the Nessler test and for ethylene oxide condensates with the cobaltothiocyanate reagent. When nitrogen was found, the residue was contaminated with fatty amide. Fatty amide was saponified by boiling the extracted residue to dryness with 10 ml. of alcoholic 1 N NaOH on a steam bath. The petroleum ether extraction was repeated to separate neutral oils from saponified amide.

Where neutral oil was contaminated with ethylene oxide condensates, the nonionic was precipitated with phosphotungstic acid according to Barber *et al.* (6). One hundred fifty milliliters of water were mixed with the extracted residue, then 10 ml. of 10% HCl and 20 ml. of 10% $BaCl_2$ were added. The solution was heated to a boil, and 20 ml. of phosphotungstic acid were added. Boiling was continued for 2 min. After cooling, the solution was extracted with petroleum ether as previously described.

Nonionics were determined by the columnar method described by Ginn and Church (3) (Step D). Owing to the larger surfactant-sample size used here, twice the amount of ion exchange resins and solvents were employed.

Anionics were eluted from the anion exchange column according to Ginn and Church (Step E). Excess sodium hydroxide was neutralized with sulfuric acid, and the solution was heated to near dryness on a steam bath. Extraction with 95% isopropyl alcohol (ASTM D1681-59T) was repeated to separate the actives from inorganic salts. Alcohol was boiled off, and the anionic residue was dried to constant weight at 60°C. in a vacuum oven.

Alkyl sulfates were hydrolyzed to their corresponding alcohols by refluxing the anionic sample for 16 hr. with 100 ml. of 2 N H_2SO_4 (Step F). The solution was neutralized with NaOH and extracted with petroleum ether to remove fatty alcohols according to the previously described method to remove neutral oils. Ether was boiled off on a steam bath, and the fatty alcohols were dried for 30 min. at 60°C. in a vacuum oven. Fatty alcohols were analyzed for total active hydrogen content by the Zerewitinoff determination.

Carboxylates (fatty acids) were recovered by repeating the petroleum ether extraction on the anionic solutions acidified to pH of 4 with sulfuric acid (Step G).

Hydrotropes were analyzed according to the method described by House and Darragh (7) (Step H). The remaining anionic solution was neutralized with sodium hydroxide, using phenolphthalein indicator, and then boiled to near dryness on a steam bath. Following this, the actives were extracted with 95% isopropyl alcohol as previously described. Isopropyl alcohol was boiled off on a steam bath, and the residue was dissolved in 100 ml. of water; 50 ml. of 9 N sulfuric acid were added. The solution was extracted with four 50-ml. volumes of ethyl ether to remove alkylaryl sulfonic acids. Combined ether layers were given three washes with 30 ml. of 3 N sulfuric acid. Aqueous layers were combined, neutralized with sodium hydroxide, and diluted with water to 700 ml. volume. Ultraviolet absorption spectrum of each solution was measured by a Cary Recording Spectrophotometer (Model 14). The amount of hydrotrope present was based on an absorption *vs.* concentration calibration curve for known compositions.

Ether was evaporated from the alkylaryl sulfonic acid solution (Step I), and the sulfonic acids were neutralized with sodium hydroxide by using a phenolphthalein indicator. Then the sulfonates were extracted with 95% isopropyl alcohol as previously described to remove inorganic salts. Next the volume of the filtrate was made up to 500 ml. with isopropyl alcohol, and a 50-ml. aliquot was analyzed for sodium chloride by ASTM D1681-59T. Then a 300-ml. aliquot was heated to near dryness on a steam bath and quantitatively transferred to a tared platinum crucible. The solvents were evaporated on a steam bath, and the active residue was dried to constant weight (± 1 mg.) at 60°C. in a vacuum oven.

The dried sample was ashed according to ASTM D1681-59T, and its combining weight was calculated. Where sodium chloride was present, it was necessary to correct for sodium chloride in the dried active residue and in the ash where the sodium chloride was converted to sodium sulfate.

Infrared absorption analyses were run on Nujol

TABLE VI
Quantitative Analysis of Actives

Ingredients	Found in detergents on anhydrous basis (%)			On "as is" basis (%)
	A	B	C[a]	D
Neutral oils	0.4 (0.3)[c] 0.4	2.0 (2.4) 2.0	0.2 0.1	0.2 (0.2) 0.2
Nonionics	0.0	3.6 (4.0) 3.7	7.8 7.8	5.1 (5.4) 5.3
Sodium dodecylbenzene sulfonate	34.3 (35.0) 34.3	10.3 (10.0) 10.1	4.3 4.4	15.0 (15.2) 14.8
Combining weight of Na DDBS	349.0 (348) 351.0	344.0 (348) 346.0	328.0 (348) 335.0	347.0 (348) 339.0
Sodium xylene sulfonate	3.1 (3.0) 3.1	2.9 (3.0) 2.8	0.0	0.0
Sulfated surfactant by difference[b]	0.0	10.4 (10.0) 10.3	0.0	11.2 (9.7) 11.6
Fatty alcohol from hydrolysis of sulfate	0.0	6.2 (7.1) 6.5	0.0	7.6 (7.8) 7.4
Fatty alcohol-active hydrogen content	.....	0.39 (0.36)	.....	0.32 (0.30)
Total actives accounted for	37.4 (38.0) 37.4	27.2 (27.0) 26.9	12.1 12.1	31.3 (30.3) 31.7

[a] Composition was difficult to drum-dry. [b] Total anionics — (Alkylarylsulfonate + xylene sulfonate). [c] Values in parentheses are theory.

mulls of the isolated nonionics, fatty alcohols from hydrolyzed alkyl sulfates, and alkylaryl sulfonates.

Discussion

Table VI lists data (in duplicate) for quantitative analysis of the actives. It is evident that the method has given good results compared to theory for neutral oils, nonionics, sodium dodecylbenzene sulfonate, and sodium xylene sulfonate contents found. Neutral oils from Detergents B and D were contaminated with fatty amides and therefore were saponified with alcoholic NaOH. The purified neutral oils were recovered, following petroleum ether extraction. Detergent C neutral oil was contaminated with fatty alcohol-EO condensate. In this case the purified neutral oils were extracted with petroleum ether, following precipitation of the nonionic with phosphotungstic acid. Amounts of contaminants removed from the neutral oils were added to the nonionics recovered from the ion exchange effluent.

Although good values were obtained for nonionic contents, in some cases the residue from the ion exchange effluent appeared to be slightly contaminated with resin. Dissolving the effluent residue in isopropyl alcohol, filtering, and evaporating the alcohol appeared to correct this error. Alkyl sulfate content was determined by difference: total anionics less the sum of alkylaryl sulfonate and hydrotrope. Good results are shown for the alkyl sulfate in Detergent B, but values for Detergent D are high. It is possible that the sample may have been contaminated with ion exchange resin at this point. Calculating the alkyl sulfate content by loss of anionic weight before and directly after hydrolysis may have provided better results. No significant amounts of carboxylates (fatty acids) were found.

Fatty alcohols from the hydrolysis were analyzed for total active hydrogen content. They are in agreement within limits of test precision with values for alcohols from the original samples. Low recovery for fatty alcohol from Detergent B may result from volatilization during drying of the extract.

Combining weights of the sodium dodecylbenzene sulfonates recovered are in good agreement with theory except those for Detergent C, which are a little low.

Infrared absorption spectra of the nonionics, sodium dodecylbenzene sulfonates, and fatty alcohols from hydrolysis of alkyl sulfates were generally comparable to spectra for the original samples of actives.

The scheme presented will provide suitable commercial analytical data for active ingredients from detergent compositions. Actives composed of mixed anionics and nonionics can be quantitatively analyzed. In case a mixture of fatty alkanolamide and ether type nonionic are recovered in the ion exchange effluent, they can be separated following saponification of the amide and extraction of the fatty acid. On the other hand, two ether type nonionics would be difficult to separate. Fatty isethionate, for example, would not be recovered intact by this scheme since it is reported to hydrolyze during elution from the anion exchange resin (3). Soaps however could be analyzed by this method.

The total elapsed time for analysis of a surfactant mixture by this scheme is about 40–60 working hours. For certain products this amount of time can be justified to obtain a complete active characterization. Once this is done, analysis of the anionic portion of similar samples could be run by cationic titration before and after acid hydrolysis to check for sulfonate and sulfate contents. Running this on the original samples of built detergents saves a considerable amount of time. The active would need to be extracted with isopropyl alcohol only for subsequent nonionic analysis by ion exchange.

Summary

A scheme has been presented for the separation of active ingredients from built detergent compositions and subsequent analysis of active components. Active components investigated are those commonly encountered in practice. Data obtained are in good agreement with expected values. The main difficulty encountered was the characterization of a fatty alcohol-EO sulfate which gave higher than expected values for its analyzed content. It was also difficult completely to characterize this fatty alcohol-EO sulfate.

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Detoxification and Deallergenization of Castor Beans[1]

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DURING THE PAST FOUR YEARS acreage planted to castor beans has increased almost five-fold, from 5.1 to 24.0 thousand acres, and the annual domestic production has increased almost fifteen-fold, from 3.4 to 49.9 million pounds (1). This is attributed to the development of high-yielding bean varieties and improved harvesting machines. These improvements have made production of this crop more attractive to farmers as a replacement for crops in surplus supply or with acreage restrictions.

A very important economic factor affecting the further development of castor beans is that the meal

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