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Use Evaluation of Alcohol Derivatives in Detergent Formulations

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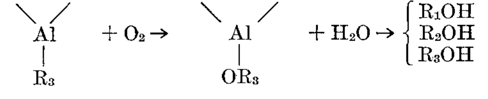
LONG CHAIN ALCOHOLS as raw materials for the detergent industry have been of significant commercial importance for many years. Due to the emphasis on biodegradability properties in today's market, the discussion here will be confined to the straight-chain primary alcohols.

These alcohols have traditionally been prepared by saponification of natural waxes, such as sperm oil, or by reduction of fatty acids obtained from oils and fats, such as coconut oil, palm kernel oil, and tallow (1). However, within the last year and one-half, straight chain primary alcohols have become available from petrochemical sources. The process for the production of these petroleum-derived alcohols (2) is briefly as follows: metallic aluminum reacts with hydrogen gas and aluminum triethyl to yield diethylaluminum hydride; the hydride is reacted with ethylene to give aluminum triethyl; the aluminum triethyl reacts with more ethylene to give a mixture of aluminum alkyls ("growth" product) containing randomly distributed alkyl groups; and this "growth" product is oxidized with air and then hydrolyzed to form even-numbered straight chain primary alcohols.



$$\overset{R_1\quad R_2}{\underset{R_3}{Al}} + O_2 \rightarrow \overset{R_2O\quad OR_2}{\underset{OR_3}{Al}} + H_2O \rightarrow \left\{\begin{array}{l} R_1OH \\ R_2OH \\ R_3OH \end{array}\right.$$

The petroleum-based alcohols and the fat and oil derived alcohols have identical physical and chemical properties when comparing the same carbon chain distributions. However, since the distribution of the petroleum-derived straight chain alcohols is governed by a Poisson distribution, proper adjustment of reaction conditions easily shifts the carbon chain length to peak at a higher or lower molecular weight. This process, therefore, can produce blends of commercial straight-chain alcohols not economically attractive in the past. Therefore, it is important to re-evaluate the effect of the carbon chain length of the alcohols upon the use properties of the major alcohol derivatives used in detergent formulations. The derivatives discussed here will be alcohol sulfates, alcohol ethoxylates and alcohol ether sulfates.

Alcohol Sulfates

The alcohol sulfates are prepared for the alcohols by typical sulfation techniques using typical sulfating agents: SO_3, cholorsulfonic acid, sulfamic acid, etc. (3). The greatest potential for alcohol sulfates in household detergent formulations appears to be in heavy duty powders. Detergency and foam stability of individual alcohol sulfates were evaluated in the following heavy duty formulation:

20%	active	15%	sodium sulfate
50%	sodium tripolyphophate	0.8%	CMC
5%	sodium silicate (meta)	9.2%	water

Foam stability tests were run in a typical plate washing test at 115F, 0.22% solids concentration, at 50 ppm hardness. In this test uniformly soiled plates are washed to obtain an end point of a permanent break in the foam covering the dishpans. One plate difference is significant below 10 plates washed. When

TABLE I
Effect of Free Alcohol on Foam Stability of Individual Alcohol Sulfates

Alcohol sulfate	Plates washed at 115F, 50 ppm hardness, 0.22% solids concentration												
	(a) As is (no free alcohol)	(b) C_{12} Alcohol per cent/active			(c) C_{14} Alcohol per cent/active			(d) C_{16} Alcohol per cent/active			(e) C_{18} Alcohol per cent/active		
		5	10	15	5	10	15	5	10	15	5	10	15
C_{12}	3	9	..	14	3	3	3	2	..	1	2	1	1
C_{14}	13	>30	..	>30*	>30	>30	>30	..	20	..	16	..	..
C_{16}	16	30	>30	>30	..	30	30	17	..	13	..	..	..
C_{18}	8–9	16	19	20	11	..	..	..	11	..	11	..	6

Note: Greater than (>) thirty plates is indicated above the 30 plate range. Due to the copious nature of the alcohol sulfate foam, it is difficult to detect an end point of this magnitude. The condition marked with an asterisk subjectively was noted to have considerable amounts of foam above the level of any others marked >30.

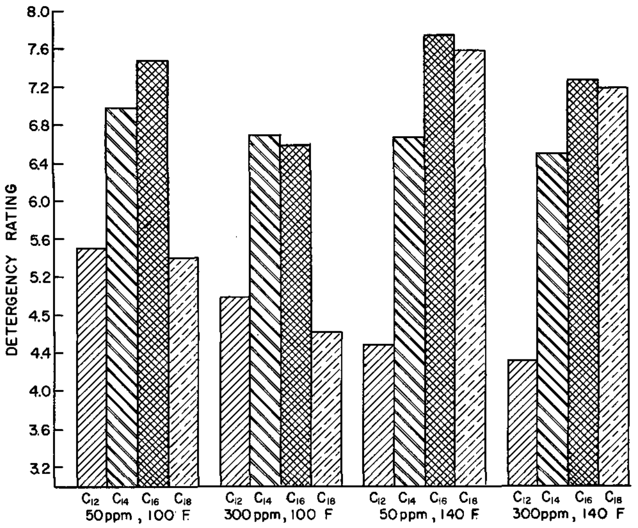


FIG. 1. Detergency of alcohol sulfates.

more than 10 plates are washed, a difference of two plates is necessary to be significant at a 95% confidence level. Table I, below, shows the effects of both molecular weight and free alcohol content upon foam stability. Individual alcohols of 12,14,16 and 18 carbon chain length were sulfated and neutralized to the sodium salt. The resulting sulfates were de-oiled and de-salted. Differing amounts of each of the alcohols were added back (on an active basis) to each of the different sulfates to determine the effect of free alcohol on foam stability.

Column (a) indicates that with pure de-salted and de-alcoholed sulfates the C_{14} and C_{16} sulfates give the superior foam stability. C_{12} alcohol (dodecyl) added back in 5, 10, or 15% amounts, on an active basis, increases the foam stability of the alcohol sulfates at all four molecular weight levels. C_{14} alcohol gives good foam stabilization of the C_{14} and C_{16} alcohol sulfates, only marginal stabilization to C_{18} sulfate, and does not affect the foam stability of the C_{12} alcohol sulfate. Familiarity with the effects makes it possible to set sulfating conditions to obtain maximum foam stability. For example, in working with lighter blends (C_{12} and C_{14}), if greater foam stability were desired sulfation could be controlled to leave 10 to 15 per cent free alcohol in the final sulfate. In working with an over-all blend (C_{12} thru C_{18}), it would be advantageous to work for a low free alcohol content and then add back small percentages of C_{12} or C_{12} and C_{14} alcohol.

The same type of study was made to ascertain effects of free alcohol on detergency. In the same manner as above, 5, 10, and 15% amounts of the different alcohols were added back to the alcohols sulfates. Over-all, there were no significant effects on detergency until large amounts (25%) of heavier alcohols (C_{16} and C_{18}) were added back to C_{12} alcohol sulfate. Under these conditions a detrimental effect on detergency was noted. Since these conditions are beyond normal expectations of amount and type of free alcohols, increased foam stability can be easily accomplished without adversely affecting detergency.

Effect of molecular weight on the detergency of alcohol sulfates was evaluated using the same experimental heavy duty formulation mentioned above. This evaluation was done on the Terg-O-Tometer at 100F and 140F, 50 and 300 ppm hardness, and 0.25% solids concentration. In this evaluation three different types of commercial standard soiled cloths were used (Testfabrics, Inc. cotton soil test cloth; American Conditioning House ACH #115 precision soiled cloth; and U. S. Testing Co. cotton standard soil cloth). Three swatches of each of the three cloths were included in each run and replicate runs were made. From the differences in reflectance between washed and unwashed cloths, a *relative* detergency rating was obtained. A difference of 1.0 in detergency rating is necessary for one sample to be indicated as better than another at a 95% confidence level. Figure 1 gives the results of this evaluation.

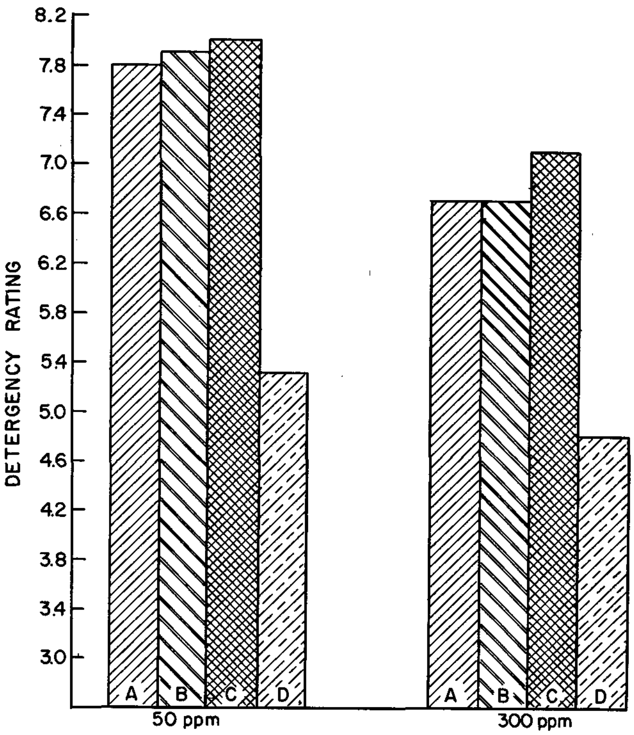


FIG. 2. Detergency of alcohol sulfate blends.

Conclusions: 1) At 100F, C_{14} and C_{16} alcohol sulfates are significantly better in detergency than C_{12} and C_{18} sulfates at both hardness levels. 2) At 140F, C_{16} and C_{18} alcohol sulfates are marginally better in detergency than C_{14} sulfate, which is, in turn, significantly better than C_{12} alcohol sulfate.

Therefore, in the over-all detergency picture at both temperature and hardness conditions the C_{16} alcohol sulfate appears to be the optimum for detergency. At high temperatures the C_{18} alcohol sulfate is as good as the C_{16} and at low temperature C_{14} is as good as the C_{16}.

The alcohol sulfates in the C_{12} to C_{18} range are excellent in detergency. Few differences in detergency among blends in this range should be expected, at average temperatures and hardness. This similarity in performance is illustrated by detergency results shown in Figure 2, where tests were run at 120F, 0.25% concentration, at 50 and 300 ppm hardness, using the same 20% active formulation mentioned before.

Code	Active ingredient (Na salts)
A	sulfate of a blend of 35% C_{16} and 65% C_{18} alcohols
B	sulfate of a blend of 65% C_{16} and 35% C_{18} alcohols
C	sulfate of a blend of C_{12} thru C_{18} alcohols
D	dodecylbenzene sulfonate (polypropylene derived)

The data from Figure 2 illustrate that there are few differences among alcohol sulfate blends in these ranges. These data also indicate, as previously reported (4), the detergency superiority of alcohol sulfates over alkylaryl sulfonates.

Foam stability tests run on the same formulations at 115F, 0.125% solids concentration, and 50 ppm hardness showed Formulation A washed 17 plates, B—18 plates, C—20 plates, and D—7 plates.

These results indicate that the optimums still hold for blends of alcohol sulfates. Formulation C (containing C_{12} thru C_{18} alcohol sulfates) should and does exhibit better foam stability since it has more of the better foaming C_{14} alcohol sulfate. The three alcohol sulfate blends compare favorably with tridecylbenzene sulfonate, which in the same formulation would wash about 18 plates.

The detergency superiority of the alcohol sulfates is shown in another manner by a comparison of several formulations containing alcohol sulfate with a typical formulation containing sodium dodecylbenzene (Na DBS) sulfonate. The following basic formulations were evaluated:

No.	% NaDBS	% alcohol sulfate	% phosphate (STPP)
1	20	0	35
2	0	7.5	50
3	0	13.5	35
4	0	10	35
5	8	9	47

In addition to these components, each formulation contained 0.8% CMC, 9.2% moisture, and was filled to 100% with sodium sulfate. Thus we will have comparisons of a typical alkylaryl sulfonate formulation (No. 1) with alcohol sulfate formulations containing less active ingredient at the same (Nos. 3 and 4) and high phosphate levels (No. 2) and a combination active product containing sulfonate and alcohol sulfate (No. 5). The alcohol sulfate used was the sulfation product of a blend of C_{12} thru C_{18} alcohols with a molecular weight of 228.

Figure 3 shows the detergency of these five formulations evaluated at three different conditions.

At normal conditions of 120F, 0.2% solids concentration, and 50 ppm hardness there are no significant differences among the five formulations, though the alcohol sulfate containing formulations are marginally better in detergency that the sulfonate formulation.

In hard water (300 ppm), but still at 0.2% concentration and 120F, the alcohol sulfate formulations are again not significantly different from each other, but are all four significantly better than the sulfonate formulation.

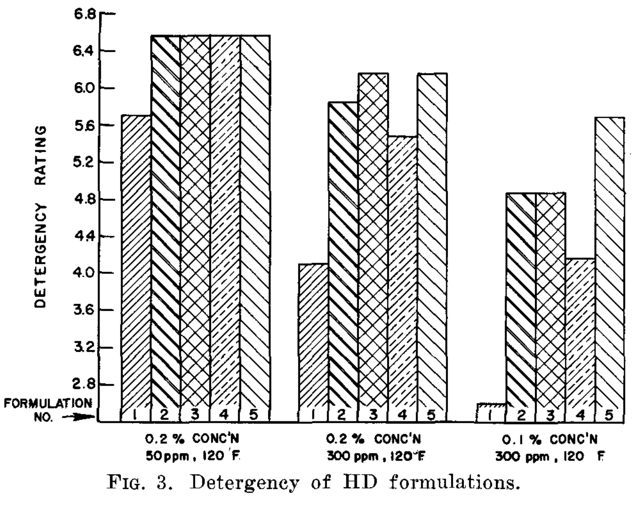


FIG. 3. Detergency of HD formulations.

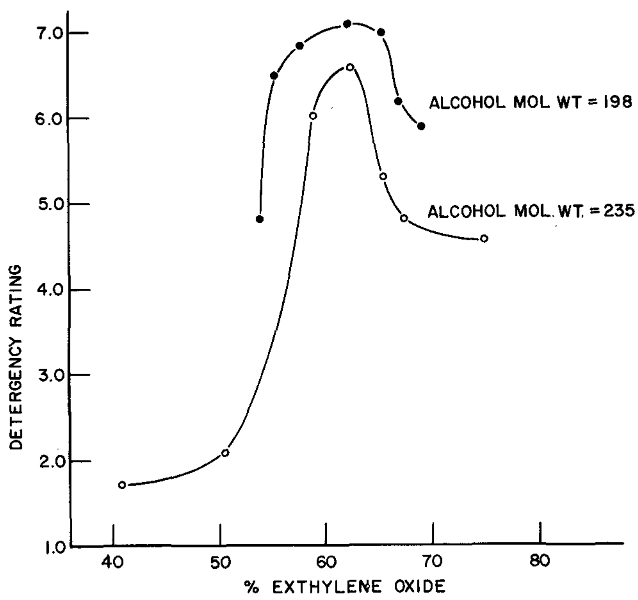


FIG. 4. Effect of ethylene oxide content on detergency.

At the very adverse conditions of below normal concentration (0.1%) and above normal hardness (300 ppm) the four alcohol sulfate formulations are still significantly better in detergency that the sulfonate formulation. Even formulation No. 4, which has only one-half the active content and is otherwise similar, is better than formulation No. 1.

Alcohol Ethoxylates

Alcohol ethoxylates are prepared by the conventional ethoxylation (5) of the alcohols resulting in the formation of a nonionic compound with extraordinary versatility. The structure of the nonionic is very flexible since the hydrophobe-hydrophile relationship within the compound can be varied by changing the ethylene oxide percentage on the alcohol or by changing the molecular weight of the alcohol. Further diversification is made possible by utilizing different distributions of individual alcohols to obtain a specified molecular weight.

The greatest potentials for the nonionics lie in the already well-developed fields of light duty liquids and low sudsing heavy duty formulations. Nonionics are used in combination with alkylbenzene sulfonates to produce high foaming light duty liquids, but in heavy duty formulations with lower active ingredient the foam is minimized. For low-sudsing or controlled-suds heavy duty formulations a major performance criterion is detergency. The effect of ethylene oxide content upon detergency is given in Figure 4. An alcohol blend made up of C_{12} and C_{14} alcohols with a blend molecular weight of 198 and a heavier alcohol blend (molecular weight—235) were ethoxylated at several different levels of ethylene oxide. These data were obtained in evaluating the same experimental heavy duty formulation mentioned previously with the entire active being represented as nonionic.

These data indicate that about 62% ethylene oxide is the optimum, regardless of hydrophobe base. Thus, for the straight-chained alcohols, it is preferable to talk of per cent ethylene oxide in the total molecule, rather than of the number of moles of ethylene oxide added to the hydrophobe. On the 198 molecular weight alcohol ethoxylate the optimum for detergency was at 62% ethylene oxide or 7.5 moles. On the higher molecular weight nonionic the optimum was also at

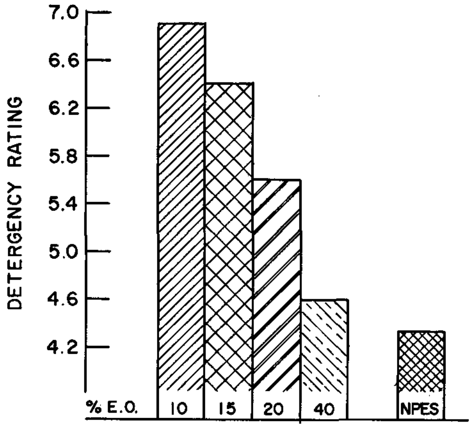


Fig. 5. Effect of ethylene oxide content on detergency of alcohol ether sulfates.

62%, but on this hydrophobe this is 9.5 moles of ethylene oxide. The same percentage optimum holds for several blends. This means that the number of moles of ethylene oxide for optimum performance will vary with the hydrophobe base, but the percentage of ethylene oxide per total weight is the same for all alcohol blends.

In low or controlled sudsing formulations it has been found that 10% nonionic is a more efficient amount of ative ingredient. Very little detergency can be gained in going above this quantity.

Nonionics in light duty liquid formulations are used in combination with alkylaryl sulfonates to give liquids with excellent foam stability. The effect of ethylene oxide on foam stability was studied in an experimental formulation consisting of 18% sodium dodecylbenzene sulfonate, 12% nonionic, and 5% lauric diethanolamide. A 198 mol wt alcohol (this is in the commercial lauryl alcohol range) was ethoxylated from 54% to 69% ethylene oxide and evaluated in the dishwashing test for foam stability. There were no significant differences among any of the samples in this range of E.O. content.

In this type of liquid formulation the cloud point of the finished formulation can be brought to less than 30F with any of the conventional hydrotropes—xylene or toluene sulfonate, ethanol, etc.

The straight chain alcohol ethoxylates in the alcohol range of C_{12} to C_{18} perform similarly in foam stability to the branch-chained tridecyl alcohol and alkylphenol ethoxylates in this type of light duty formulation.

Alcohol Ether Sulfates

The alcohol ether sulfates are prepared by the sulfation of the alcohol ethoxylate in a manner similar to the sulfation of the alcohol itself. One major advantage of the alcohol ether sulfates over alkylphenol ethoxylates is the ability to sulfate with SO_3, chlorosulfonic acid, or a variety of sulfating agents, since ring sulfonation is not a factor. This, in turn, gives diversity in choice of the cation salt resulting from neutralization of the sulfation product.

The alcohol ether sulfates have potential in both heavy duty and light duty liquid formulations. The effect of ethylene oxide content upon detergency in a heavy duty formulation is shown in Figure 5.

Ethylene oxide adducts containing from 10% to 40% E.O. were prepared using an alcohol containing C_{12} thru C_{18} alcohols. Detergency evaluations were made at 140F, 0.25% concentration at 50 and 300 ppm hardness. There were no significant sample-hardness interactions; therefore, the detergency ratings shown in Figure 5 are derived from the totals of the two hardness levels.

TABLE II

Effect of E.O. Content on Foam Stability and Cloud Point of LD DB/ES Liquids

ES portion of active (sodium salts)	Plates washed at 115F, 0.05% concn.		Cloud point °F
	50 ppm	300 ppm	
198 mol wt alcohol w/17% E.O.[a, b]	31	34	48
198 mol wt alcohol w/33% E.O.	30	32	34
198 mol wt alcohol w/40% E.O.	30	32	30
Nonylphenol ES w/44% E.O.	26	30	54
Tridecyl alcohol ES w/40% E.O.	25	29	60

[a] The alcohol used here is 55% C_{12} and 45% C_{14}.

[b] % E.O. refers to per cent ethylene oxide per total weight of the ethoxylate before sulfation.

These data indicate that as the ethylene oxide content is decreased the detergency is increased. This would be expected. As the ethylene oxide is decreased the alcohol sulfate is approached; and as was already discussed, the alcohol sulfate is an excellent detergent. Figure 5 also indicates only a marginal advantage in detergency for the alcohol ether sulfates at 40% E.O. over a conventional alkylphenol ether sulfate (NPES). Lower E.O. contents are significantly better than NPES in detergency.

In light duty liquid formulations alcohol ether sulfates can be used in combination with alkylaryl sulfonates or as the sole active ingredient. A study of the ethylene oxide content of the ether sulfate on the foam stability of a dodecylbenzene sulfonate—ether sulfate (DB/ES) formulation is given in Table II. The experimental formulation is 18% sodium DB, 12% sodium ES, 5% lauric diethanolamide, 10% ethanol, 55%water.

There is no significant effect on LD liquid foam stability at either hardness level due to the ethylene oxide content of the ether sulfate in a DB/ES formulation. The alcohol ether sulfate is better than the nonylphenol and tridecyl alcohol ether sulfates at normal hardness. At high hardness, as is classically true, the branch chain improves relative to the straight chain and only a marginal difference is noted between the alcohol and the branch-chained products.

The effect of ethylene oxide content on the solubility of LD liquid DB/ES formulations is indicated by the cloud point of the formulation also shown in Table II.

Solubility is decreased proportionally as ethylene oxide content is lowered. The straight chain alcohol ether sulfates at all levels of ethoxylation evaluated impart more solubility to this formulation than branched ether sulfates.

The other type of light duty liquid formulation investigated contained ether sulfate as the sole active ingredient. Only straight chain alcohols were included in this study, as these are the only alcohols used in this type of formulation in the U.S. This formulation was 25% ether sulfate, 5% lauric diethanolamide, 10% ethanol, 60% water. The effect of ethylene oxide content on foam stability in this formulation is shown in Table III.

TABLE III

Effect of E.O. Content on Foam Stability of LD ES Type Liquids

ES portion of active (Na salt)	Plates washed, 115F, 0.05% concn., 50 ppm hardness
198 mol wt alcohol w/17% E.O.	25
198 mol wt alcohol w/33% E.O.	20
198 mol wt alcohol w/40% E.O.	17
Natural derived lauryl alcohol w/40% E.O.	16

TABLE IV

Effect of Alcohol mol wt on Foam Stability of DB/ES Liquids

ES portion	% E.O.	Plates washed, 115F, 50 ppm, 0.05% concn.
C_{12}	40	31
C_{14}	40	29
C_{16}	40	20
C_{18}	40	10

TABLE V

Foam Stability Performance of Ether Sulfates of Alcohol Blends (DB/ES formulation)

Alcohol distribution				% E.O.	Plates washed at 115F, 0.05% concn.	
C_{12}	C_{14}	C_{16}	C_{18}		50 ppm	300 ppm
55	45	....	....	40	30	32
62	26	12	....	40	30	32
40	30	20	10	40	28	32
....	....	65	35	40	17	....
Nonylphenol				40	26	30

These data indicate that as the ethylene oxide content is decreased the foam stability increases. Again, as with detergency, this increase in performance occurs as the alcohol sulfate is approached. At equal ethylene oxide percentages there are no significant differences in foam stability with this formulation between straight-chain petroleum-derived and naturally-derived alcohol ether sulfates.

In light duty liquids the optimum chain length of the alcohol ether sulfates for foam stability is C_{12}, with C_{14} close behind as indicated by data on the DB/ES formulation given in Table IV.

Though the foam stability performance drops at C_{16} and C_{18}, small percentages of these heavier alcohols do not inhibit the effectiveness of more economical broad blends of the alcohols (see Table V).

The straight chain alcohol ether sulfates in light duty liquids have advantages over branched-chain ether sulfates in foam stability and solubility in addition to being biodegradable.

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Recent Advances in Fatty Amine Oxides. Part II. Formulation and Use

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ALKYLDIMETHYLAMINE OXIDES as foam stabilizers for detergent formulation have gained considerable interest the last few years. However, amine oxides are excellent detergents in their own right as the sole active ingredient (1,2). For example, in a 20% active heavy duty formulation dodecyldimethylamine oxide (C_{12} DMAO) was compared with alkylaryl sulfonates in foam stability and detergency at 50 and 300 ppm hardness (Table I).

C_{12} DMAO exhibits foam stability between that of dodecyl and tridecylbenzene sulfonates. The detergency of C_{12} DMAO is similar to that of the alkylaryl sulfonates at both hardness levels.

Due to the economics of production, the practical interest in the amine oxides is primarily in the field of foam stabilization, rather than as a major portion of the active content. The amine oxides have been reported as excellent foam stabilizers for light duty liquids (3).

Light Duty Liquids

Light duty liquids in this country are made up of three major types based on their active ingredient: 1) DB/ES type—a blend of alkylaryl sulfonate, usually dodecyl, with an ether sulfate (the sulfate of an ethoxylated alcohol or alkylphenol); 2) DB/NI type—a blend of alkylaryl sulfonate and a nonionic (the ethoxylate of an alcohol or alkylphenol); and 3) Straight ES type—the sole active ingredient is an alcohol ether sulfate.

TABLE I

Active ingredient	Plates washed [a] 115F, 0.125% concn.		Detergency rating [b] 120F, 0.2% concn.	
	50 ppm	300 ppm	50 ppm	300 ppm
C_{12} DMAO	13	19	6.0	5.1
Na dodecylbenzene sulfonate	5	18	5.7	4.4
Na tridecylbenzene sulfonate	17	25	6.3	4.9

[a] Number of uniformly soiled plates washed to obtain an end point of a permanent break in the foam covering the dishpan. Two plates difference is necessary in order to be significant at a 95% confidence level.

[b] One unit difference in detergency rating is necessary in order to be significant (95% confidence). Detergency ratings are relative, rather than absolute, and are obtained from the differences in reflectance between washed and unwashed standard soiled cloths washed in a Terg-O-Tometer.

TABLE II

Formulation	Foam stabilizer	Δ P, difference in plates washed compared with LDEA [a] at 115F, 50 ppm hardness, 0.05% concn.
DB/ES	C_{12} DMAO	−4
DB/NI	C_{12} DMAO	+1
ES	C_{12} DMAO	+6

[a] A difference of two plates is necessary in order to be significant at 95% confidence level.

Amine oxides were evaluated as foam stabilizers in each of these types of formulations. The actual formulations were:

DB/ES 18% sodium DB sulfonate, 12% sodium lauryl ether sulfate, 5% foam stabilizer
DB/NI 18% sodium DB sulfonate, 12% lauryl alcohol ethoxylate (62.5% E.O.), 5% foam stabilizer
ES 25% sodium lauryl ether sulfate (3 mole ethylene oxide adduct), 5% foam stabilizer

Cursory foam stability evaluations of each of these formulations comparing C_{12} DMAO with LDEA (lauric diethanolamide) are give in Table II.

These results indicate that the best potential for the amine oxides in light duty liquids was in the ES type formulation.

The effect of molecular weight on foam stability was studied to determine optimum carbon chain length. C_{10} through C_{18} DMAO were prepared from the appropriate petroleum-derived straight chain alcohols and compared with LDEA (Table III).

The optimum DMAO for foam stability in this