A Novel Dianionic Surfactant from the Reaction of C₁₄-Alkenylsuccinic Anhydride with Sodium Isethionate¹

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A novel derivative of alkenylsuccinic anhydride has been developed. When the anhydride is opened with sodium isethionate, a difunctional surfactant, alkenyl carboxysulfonate (ACS), is produced. This product has a unique combination of properties: moderate foaming, effective detergency, as well as the capability to function as a hydrotrope and as a co-builder in formulated cleaning systems. This paper briefly reviews some past studies with ACS. The utility of ACS in hard-surface cleaning is also examined, especially the capacity of this molecule to act both as a low-streaking surfactant and a hydrotrope. This combined function should allow formulators to diminish or eliminate volatile solvents in a variety of cleaning products. ACS has shown merit as an agent to reduce soil redeposition in three different heavy-duty liquid formulations. Additionally, heavy-duty liquid detergents containing ACS can be formulated to high surfactant and organic builder levels.

KEY WORDS: Anionic, carboxysulfonate, co-builder, detergent, hydrotrope, redeposition, surfactant.

Information on the synthesis and applications of surfactant derivatives of alkenylsuccinic anhydride (ASA) has been published previously (1). These developmental surfactants have been designated as alkenyl carboxysulfonates (ACS). This nomenclature highlights that these are difunctional derivatives of ASA, having both carboxylate and sulfonate anionic groups.

The prior publication (1) described two possible products, an isethionate and an N-methyltaurate. Process studies have caused us to focus on the isethionate derivative. Past applications studies have shown that ACS is an effective detergent and an exceptional co-builder, and it offers substantial efficacy as a hydrotrope. This paper briefly reviews some of this past work, and emphasizes how ACS could fit particularly well into hard-surface cleaners and heavy-duty liquid laundry products.

A possible view of the future for formulators is that each ingredient will have to be efficient. Thus, a surfactant that also functions as a hydrotrope will have great value and utility. ACS is such a molecule.

EXPERIMENTAL PROCEDURES

The ACS samples used in this research were synthesized at Shell (Houston, TX) or by a contractor by means of a proprietary process. Except for the comparison of the stability of ACS N-methyltaurate to ACS isethionate, all ACS used in this research was the ACS isethionate. All intermediates and products were characterized by typical methods, including ¹³C nuclear magnetic resonance. The comparison surfactants were obtained from a variety of sources and are identified later by both chemical description and trade name. Builders, caustic and other laboratory chemicals were obtained from standard commercial sources and used as received. All solution concentrations are given in percentage by weight of the solute in deionized water.

Surfactant activity was determined with the ORION 9342BN electrode (surfactant-specific) and an ORION 93-02 double-junction reference electrode (Orion Research Inc., Boston, MA). Hyamine 1622 (Orion Research Inc.) was the cationic titrant, and a Metrohm E536 titrator (Brinkmann Instruments, Houston, TX) was used. Procedures for radiotracer detergency have been published (2,3). For the hydrotrope studies, iterative additions of hydrotrope were made, along with water, so that the overall formulation concentration remained constant. Dynamic surface tension was measured with the maximum bubble pressure method on a Sensadyne device (Chem-Dyne Research Corp., Mesa, AZ). For foaming, a volume of surfactant solution was shaken twenty times in a graduated cylinder at atmospheric pressure. The pH adjustments were made with dilute caustic or sulfuric acid. All viscosity data were determined with a digital Brookfield viscometer (Model LVT; Brookfield Engineering Laboratories, Inc., Stoughton, MA). For the calcium inhibition measurements, a commercial dipping-probe colorimeter was immersed into the aqueous solution, which was agitated with a nonaerating stirrer. Solution transmittance was monitored with time. These experiments have been described (4,5).

To compare surfactant streaking, aqueous 1% (wt) solutions are wiped across solvent-cleaned black tiles, with the Gardner in-line hard-surface cleaning device (Bethseda, MA). The usual cleaning sponge is covered with a paper towel to absorb the test surfactant. The tiles were air-dried after 30 wiping strokes. The change in "Y" reflectance of the black tile is a relative measure of the residual surfactant. Reflectance differences of approximately 0.1 are statistically significant.

For reflectance detergency and redeposition values, EMPA 101 fabric (Testfabrics, Inc., Middlesex, NJ) was washed with two commercial liquid detergents along with a laboratory-formulated heavy-duty liquid (HDL), which contains linear alkylbenzenesulfonate (LAS) and AEO 25-7. Cloth reflectance was determined with a Hunter Labscan 6000 spectrocolorimeter (Hunter Associates Laboratory, Inc., Reston, VA), which was used to measure either "Y" or "WI" reflectance. Redeposition and cleaning were determined in a single Terg-O-Tometer experiment, with a clean swatch added to allow redeposition. The values were calculated as follows:

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[%] soil removal = [Y washed - Y soiled/Y unsoiled - Y soiled] \times 100 % redeposition = ["WI" unwashed - "WI" washed/"WI" unwashed] \times 100 [1]

The reported formulation stability results were obtained by making concentrated slurries of the surfactants and builder (sodium citrate) at the indicated ratios. These dispersed blends were diluted with water or the indicated combination of water and hydrotrope. More water or mixed solvent was added until the formulation was both optically clear and fluid.

The SKINTEXTM and EYTEX[®] assays were commercially available from In Vitro International (previously Ropak Laboratories; Irvine, CA). For this work, pH-adjusted aqueous solutions were sent to the laboratory for evaluation. The test materials were identified only by number. An overview of these methods has been published (6).

Synthesis. The molecular structure, which is referred to in this paper as ACS, is synthesized according to Scheme 1. The synthesis begins with a thermal "ene" reaction (7) between an alpha-olefin and a maleic anhydride. The resulting ASA is an example of a commonly used paper sizing agent (8). Previous work has shown that the most useful ASA for subsequent derivatization to a surfactant (9) is based on C_{14} alpha-olefin. Shorter-chain olefins yield surfactants with high critical micelle concentrations (CMC), whereas higher-molecular weight starting olefins give products with inadequate water solubility. The second step in the production of ACS involves the thermal opening of the anhydride ring with sodium isethionate. Recent advances in the method of synthesis have yielded a product with chemical purity of greater than 95%. The typical material is a slightly hygroscopic solid.

For performance and synthesis reasons, we focused on the isethionate derivative of ASA. Sodium N-methyltaurate is substantially more costly than sodium isethionate and is also much more hygroscopic than the isethionate. This more hygroscopic nature makes handling more difficult, especially for operation on a large scale. Further, there are only insignificant differences in performance between the two derivatives. It was initially believed that the amide would be less subject to hydrolysis than the isethionate. This is indeed the case; however, as will be discussed, the isethionate is quite stable, especially within the pH range of typical liquid detergent products. Table 1 lists various chemicals and their trade names, Table 2 contains some typical properties of ACS.

Review of previous results. As reported by Wood and Lilienthal in 1989 (unpublished results), ACS can be an ef-



SCHEME 1

TABLE 1

Identification of Chemicals^a

Chemical description	Trade name	Acronym	
C _{12,13,14,15} alcohol ethoxylate-9 EO	NEODOL® 25-9 ^b	AEO 25-9	
C _{12,13} alcohol ethoxylate-5 EO	NEODOL 23-5 ^b	AEO 23-5	
C _{12,13} alcohol ethoxylate-6.5 EO	NEODOL 23-6.5 ^b	AEO 23-6.5	
$C_{12,13,14,15}$ alcohol ethoxysulfate, sodium salt	NEODOL 25-3S ^b	AEOS	
C _{9,10,11} alcohol ethoxylate-6 EO	NEODOL 91-6 ^b	AEO 91-6	
C _{12,13,14,15} alcohol ethoxylate-7 EO	NEODOL 25-7 ^b	AEO 25-7	
Sodium C _{12,15} Pareth-6-carboxylate	Sandopan [®] DTC ^c	AEOC	
Alkyl polyglycoside	Henkel APG [®] 350^d	APG	
Dicarboxylic acid	DIACID H-240 ^e	DIACID® H-240	
C_{12} linear alkylbenzene sulfonate	Witconate® 1260 ^f	C ₁₂ -LAS	
\mathbf{C}_{11} linear alkylbenzene sulfonate	Witconate® 1160 ^f	C ₁₁ -LAS	
Ethoxy phosphate	TRITON® H-66 ^g	H-66	
Propylene glycol	Aldrich reagent ^{h}	PG	
Sodium dioctyl sulfosuccinate	AEROSOL® OT ⁱ	SDS	
Paraffin sulfonate	HOSTAPUR [®] SAS ^j	Para. Sulfonate	
Sodium dodecyl diphenyloxide disulfonate	Dowfax® 2A1 ^k	Dowfax 2A1	
Sodium cocoyl isethionate	IGEPON® AC-78 ^l	SCI	
Sodium xylene sulfonate	Witconate® SXS ^f (40%)	SXS	
Triethanolamine	Aldrich reagent ^{h}	TEA	
Monoethanolamine	Aldrich reagent ^h	MEA	
$\overline{^{a}}$ EO, ethylene oxide.			

^bShell Chemical Co., Houston, TX. ^cSandoz Chemicals, Charlotte, NC ^dHenkel Chemicals, Cincinnati, OH. ^eWestvasco Co., Mulberry, FL. Witco Co., New York, NY. ^gUnion Carbide Co., Danbury, CT. ^hAldrich Chemical, Milwaukee, WI. ⁱCytec (American Cyanamid) West Patterson, NJ. ^jHoechst-Celanese, Charlotte, NC. Dow Chemical, Midland, MI. ^lRhône-Poulenc, Cranberry, NJ.

fective detergent and an exceptional co-builder, and this molecule functions as a hydrotrope in certain formulations.

Figure 1 displays the stability of ACS isethionate and ACS N-methyltaurate at 25°C. As shown in the graph, ACS isethionate loses less than 7% of its surfactant activity over twelve weeks. As noted in the Experimental

TABLE 2

Selected Alkenyl (Carboxysulfonates Properties
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Color	Tan to yellow
Physical form	hygroscopic solid
Viscosity of a 10% solution (cps)	>1500 (pH = 5) 100-200 (pH = 8) Slightly
pH of 1% solution	4.5
Critical micelle concentration (% wt)	0.01

Procedures section, the surfactants were determined with a surfactant electrode to titrate only the sulfonate and not the carboxylate group in ACS. ACS N-methyltaurate retained slightly more activity over the same time period. However, it has been concluded that this minor difference is not large enough to justify the added difficulty of synthesizing the amide derivative.

Figure 2 exemplifies a radiolabelled detergency study with prototype HDL formulations. Three different combinations of soil and fabric were used in this comparison of soil removal (2,3). ACS gives better performance than C_{12} -LAS in every comparison. Significantly, however, ACS performs nearly as well as AEO 25-9 in removing multisebum soil. Thus, ACS offers a broad range of applicability in HDL formulations. It was observed in these studies that ACS assisted in the stabilization of these formulations (9).

Figure 3 exhibits the utility of ACS as a co-builder in a heavy-duty powder formulation. The formulations contained AEO 23-5 as the sole surfactant, with either sodium tripolyphosphate, zeolite or sodium carbonate as the builder. In a second set of experiments, the builder was reduced from 30 to 5%, and 5% of ACS was used as a co-builder. As shown in Figure 3, ACS is particularly



FIG. 1. Alkenyl carboxysulfonates (ACS) stability study (12 wk) of dilute solutions at alkaline pH.

effective with both carbonate and zeolite in enhancing clay soil detergency (9). These results were obtained with insitu formulations; that is, the various ingredients were mixed in the Terg-O-Tometer. Separate evaluations have shown, however, that ACS may be agglomerated onto powders by dissolving it in a nonionic surfactant.

Results that show the effectiveness of ACS as a hydrotrope for a prototype hard-surface cleaner (HSC) are displayed in Figure 4. With four different anionic surfactants, LAS, a primary alcohol sulfate, a paraffin sulfonate, and an alcohol ethoxysulfate (AEOS), ACS showed superior ability to clarify this formulation. This benefit should allow formulators to make more concentrated and more effective products.

RESULTS

pH Effects. As the research with ACS has continued, it has been attempted to elaborate on the past advantages, while also evaluating new applications areas. Because



FIG. 2. Heavy-duty liquid laundry detergency (40°C at 150 ppm water hardness). LAS, linear alkylbenzenesulfonate; TEA, triethanolamine; PE/C, 65% polyester/35% cotton fabric. See Figure 1 for other abbreviation.



FIG. 3. Co-builder effect of ACS in laundry powders (clay soil on PE/C, 40°C, 150 ppm water hardness). Abbreviations as in Figures 1 and 2 and STPP, sodium tripolyphosphate.



FIG. 4. Hydrotrope comparisons in a built hard-surface cleaner. Abbreviations as in Figures 1, 2 and 3; SXS, sodium xylene sulfonate; AL, aluminum; PARA, paraffin.



FIG. 5. Alkenyl carboxysulfate surface tension/foam height vs. pH (dynamic surface tension at 1 bubble/s).

ACS is difunctional, surfactant properties were investigated as a function of pH. The variation of surface tension and foaming with pH is displayed in Figure 5. Surface tension was evaluated with a dynamic method, and foaming was determined by a shake foam procedure. As illustrated in Figure 5, both the surface tension and foaming show sharp changes as the pH is adjusted. When the pH was raised, the carboxylic acid group was converted to a carboxylate anion. It is presumed that this change makes ACS more water-soluble, thus raising the surface tension and dramatically reducing foaming.

Control experiments showed that the variation of surface tension with pH was reversible. For example, at pH 8, dilute sulfuric acid could be added dropwise, and the surface tension fell with the reduction in pH. After reaching a pH of approximately 4, the solution pH was raised with dilute NaOH. The values of surface tension and pH were easily reproduced. These results were interpreted as showing that ACS is stable over the time of these reactions (and over this pH range). Thus, the variations in properties seen in Figure 5 are believed to result from the changes in the ionization of the surfactant, and not to have been caused by any decomposition effects.

Not surprisingly, the solution viscosity for ACS varies with pH. Because ACS is made from sodium isethionate, the immediately formed product contains half of the sodium ions required to fully ionize ACS. As shown in Figure 6, a 10% (by weight) solution of ACS shows a dramatic drop in viscosity at pH 5.0 (when the pH is raised with NaOH). This is attributed to the change in solubility of ACS as the carboxylate anion is formed. A 33% activematter solution of ACS is easily pourable when ACS is neutralized with NaOH to a pH of approximately 8. Higher concentrations with only sodium ions are gels. Alternatively, when KOH is used for pH adjustment, a 42% solution is pourable.

No efforts have been made to date to look for hydrotropes to solubilize ACS. Experiments to make more concentrated aqueous solutions of ACS have focused on organic bases to fully neutralize ACS. When ACS is neutralized to a pH of approximately 8.5 with monoethanolamine, a 57% active-matter solution can be prepared.

Inhibition of calcium ion precipitation. Another striking feature of ACS is the interaction with calcium ions (Fig. 7). ACS was mixed with calcium nitrate, and the soluion turbidity was monitored over time with a dipping probe colorimeter. In Figure 7, ACS inhibits the precipita-



FIG. 6. Viscosity vs. pH for 10% alkenyl carboxysulfonate.



FIG. 7. Inhibition of calcium precipitation by alkenyl carboxysulfonate (0.1%) with 4% C^{++} .



FIG. 8. Inhibition of calcium precipitation by alkenyl carboxysulfonate (0.1%) at pH 10 with 4% C⁺⁺.

tion of calcium, with the best inhibition at pH 8, which is a common buffer point for HDLs. Even when the inhibition is poorest (at pH 11), little turbidity is developed in these solutions for more than one hour. This suggests that calcium would not interfere with typical household washing operations, where the washing cycle time is usually less than twenty minutes.

Figure 8 shows that at pH 10 there is a temperature



FIG. 9. Pine oil cleaner formulability for alkenyl carboxysulfonate and competitive hydrotropes. Abbreviations as in Figures 2 and 4; IPA, isopropyl alcohol.

effect—Calcium precipitates faster at higher temperatures. However, even under the least favorable circumstances (pH 11 and high temperature), insignificant turbidity develops over approximately twenty minutes. ACS thus seems suitable for use as a household washing additive, especially as typical washing temperatures are declining.

An additional possible utility of this inhibition of calcium precipitation would be the impact on waste-water treatment. Instead of chelating the calcium, as is done by certain phosphates and organic compounds (for example, ethylenediaminetetraacetic acid), the calcium ions are suspended for a relatively brief time. In this way, it is presumed that the calcium and ACS would precipitate at the water treatment site, rather than being released into the receiving waters (along with the suspending agent).

HSC. As previous work had shown the value of ACS as a hydrotrope for HSC, this applications area was investigated further. Because a major variety of HSCs utilizes pine oil, the efficacy of ACS as a hydrotrope in this category was evaluated (Fig. 9). ACS has a remarkable ability to stabilize this particular formulation. This ACS-containing formulation is believed to be a microemulsion. Further, all of these cleaners gave a substantial "bloom" when the formulation was diluted with water. This white, blooming cloud is believed to result from the disruption of the microemulsion droplets. When the microemulsion reforms into an emulsion, the larger droplets refract light, giving the characteristic white color. Consumers seem to equate an intense bloom to excellent cleaning power. One substantial advantage for the ACS formulation, especially when compared to isopropyl alcohol, is that the resulting formulation has significantly less volatile solvent. This formulation change would be expected to lead to less volatile organic carbon emissions.

An important criterion for selecting surfactants for HSC is the streaking on the cleaned surface. This phenomenon was evaluated with black tiles, a method developed at Shell (Fig. 10). ACS has equivalent low streaking to two widely recognized low-streaking surfactants, an APG (10)



FIG. 10. Streaking of 1% surfactant solutions on black ceramic tile. Abbreviations as in Figures 1 and 2; SCI, sodium cocoyl isethionate; SDS, sodium dioctyl sulfosuccinate; AEOS, alcohol ethoxylate; AEOC, alcohol ethoxycarboxylate; APG, alkyl polyglycoside.

and a carboxylated nonionic [or alcohol ethoxycarboxylate (AEOC)]. In comparing ACS to sodium cocoyl isethionate, it was concluded that the advantage for ACS is not simply the presence of the isethionate group. Similarly, a sulfosuccinate exhibits greater streaking than ACS. It is surmised that, for ACS, the low streaking results from the difunctional character of this surfactant. It is interesting that a typical 3-mole ethoxysulfate (AEOS) also gave low streaking.

HDLs. Evidence of the ability of ACS to reduce clay soil redeposition was discussed in a previous publication (1). Having found that ACS offers several advantages for



One Cycle, 500 ppm Hardness, 40°C

FIG. 11. Reflectance clay detergency and redeposition of EMPA clay (Testfabrics, Middlesex, NJ) on 65/35 PE/C. Abbreviations as in Figure 1; HDL, heavy-duty liquid (detergent).

HSC, ACS was evaluated for use in HDLs. ACS was screened for redeposition, and a reflectance method was used to evaluate both detergency and redeposition. As depicted in Figure 11, a prototype HDL was evaluated, along with two different commercially available products. In this study, for each formulation ACS raised the detergency of the formulation while reducing the amount of soil redeposited on a clean swatch. These were encouraging results, and the follow-up has been to evaluate the utility of ACS in formulating concentrated HDLs. Overall detergency results were low in all experiments; we concluded that the relatively high hardness level in these determinations significantly reduced the cleaning efficiency of all formulations.

Shown in Figure 12 are representative results of substituting ACS for part of the C_{12} -LAS and AEOS in a concentrated HDL (approximately 1/3 cup). As indicated there, a starting HDL formulation was made that include nonionic and anionic surfactants, sodium citrate and a common hydrotrope [sodium xylenesulfonate (SXS) or propylene glycol (PG)]. Water was added in increments until the formulations were clear and easily pourable at room temperature.

The ACS-containing formulation with SXS achieved fluidity at a substantially higher total surfactant and sodium citrate concentration than a comparison formulation without ACS (Fig. 12). Complementary results were obtained with the same overall formulation, but with PG substituted for SXS. This resulted in HDLs with either PG or SXS that require only approximately 40% water to be both clear and pourable. These formulations have approximately 10% each of AEO 23-6.5, C_{12} -LAS, AEOS and ACS. Additionally, each formulation contains approximately 12% sodium citrate builder. No attempts were made to add minor ingredients, such as dyes or optical brighteners.

These studies were extended to a similar formulation that alternatively used C_{11} -LAS, (Fig. 13). As in the previous cases, the starting formulation had a 3:1



FIG. 12. ACS effect on the concentration of surfactants (including C_{12} ·LAS) and citrate in heavy-duty liquids. PG, propylene glycol; MEA, monoethanol amine. Other abbreviations as in Figures 1, 2, 4 and 10.

anionic/nonionic ratio, with AEOS and the C_{11} -LAS as the anionic components. This formulation was modified to include ACS as one of the anionic surfactants, and all formulations had sodium citrate builder. Without ACS or a hydrotrope, the maximum total surfactant concentration was 21%. However, when ACS was substituted for a portion of both the C_{11} -LAS and AEOS, the total surfactant level in the clear, pourable product was 34% without any hydrotrope and 40% when SXS was included. When C_{11} -LAS was used, the combination of ACS with either SXS or PG allowed the production of a clear, flowable HDL containing approximately 40% surfactant and 12%

$Surfactant^b$	Scores		Descriptors	
	SKINTEX ^c	$EYTEX^d$	SKINTEX	EYTEX
ACS	1.53	9.6	Mild	Minimal
AEOS	0.08	10.3	Minimal	Minimally irritating
C ₁₂ -LAS	1.9	22.8	Mild	Mildly/moderately irritating
AEO 25-7	0.25	4.4	Minimal	Minimal

 TABLE 3

 Skin and Eye Irritation by in vitro Assay^a

^aSurfactants tested at 1 wt% in water.

^bACS, alkenyl carboxysulfonate; AEOS, alcohol ethoxysulfate; LAS, linear alkylsulfonate.

^cIn the SKINTEXTM assay, surfactants are classified into four classes: minimal (<0.5), mild (0.6-2.0), moderate (2.1-5.0) and severe (5.1-8.0). The scores are Primary Dermal Irritation Index, and have been correlated to past studies (11).

 d In the EYTEX® assay, surfactants are classified into six classes: minimal (0–110), minimally irritating (>10–17), mildly irritating (>17–22), moderately irritating (>22–35), severely irritating (>35–50) and extremely irritating (\diamond 50). The scale is 0–110 (12).



FIG. 13. ACS effect on the concentration of surfactants (including C_{11} -LAS) and citrate in heavy-duty liquids. Abbreviations as in Figures 1, 2, 4 and 12.

sodium citrate. These results offer the formulator options of surfactant and hydrotrope to make a variety of citratebuilt liquid detergent products.

Thus, with C_{11} -LAS-based formulations, the inclusion of ACS (without hydrotrope) leads to an approximate 50% increase in the surfactant concentration in an HDL. Further, the percentage increase in sodium citrate concentration is 60%. Substantial percentage increases are also observed when ACS is used in the presence of SXS and PG. These prototype formulations are exactly in line with the trend to more concentrated liquid formulations. Highly concentrated products of this type lead directly to reduced shipping costs for the manufacturer of the HDL, as well as less waste disposal for the consumer.

Predicted skin and eye irritation. A wide variety of possible uses for ACS have been considered, from hard-surface cleaning to liquid or powder laundry products. A possible extension of ACS applications would be in the general area of personal care-products. To provide guidance for personal care applications, *in vitro* assays were performed to assess the possibility of irritation to both skin and eye with ACS. Two commercially available assays, the SKINTEXTM and EYTEX[®], were chosen to survey how compatible ACS might be with use in a personal-care product.

Data for ACS and representative comparison surfactants are displayed in Table 3. These data suggest that ACS should be less irritating (11) to the skin than LAS, but somewhat more irritating than AEOS. A representative alcohol ethoxylate, AEO 25-7, was included for further reference. A somewhat different order of eye irritation is predicted by the EYTEX (12,13) method, where ACS is assayed as more comparable to AEO 25-7 than to either AEOS or C_{12} -LAS. The nonionic was predicted to have very low irritation potential.

Implications for future formulations. ACS has excellent surfactant properties, including a relatively low CMC, moderate foaming, low streaking and good solution stability. ACS is an effective cleaner, both for hard surfaces and for laundry applications. For laundry uses, ACS is a potent agent to prevent soil redeposition during the cleaning operation, to inhibit the precipitation of calcium and to facilitate the effectiveness of common builders (carbonate and/or zeolite or phosphate salts). Perhaps most importantly, ACS assists in the stabilization of liquid formulations. Thus, concentrated prototype products have been made where ACS functions both as a cleaner and a hydrotrope.

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