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3,492,239

LIGHT-COLORED SULFONATION PRODUCTS

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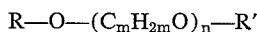
Int. Cl. C11d 1/12; C07c 143/16

U.S. Cl. 252-161

10 Claims

ABSTRACT OF THE DISCLOSURE

Process for preparing surface-active sulfonates comprising reacting an olefin having 8 to 22 carbon atoms in its molecule with at least one member selected from the group of chlorosulfonic acid and gaseous sulfur trioxide diluted with an inert gas as sulfonating agent, in the presence of a polyglycol ether having the formula:



wherein R represents alkyl of 8 to 22 carbon atoms, R' represents a member selected from the group of hydrogen and alkyl having 1 to 6 carbon atoms, *m* is 2 or 3 and *n* is a whole number from 1 to 20 and thereafter hydrolyzing the sulfonated olefin product thereby produced. The olefin sulfonates as thereby obtained are characterized by their light color and by the low percentage of non-surfactant components.

The present invention relates to the sulfonation of olefins to produce light-colored olefin sulfonates having a low content of non-surfactant components.

It has been proposed in the prior art to sulfonate olefins which have a terminal double bond by treating them with gaseous sulfur trioxide diluted with an inert gas, and to convert the sulfonated products thus produced by hydrolysis into water-soluble sulfonates. As in this process, a high degree of sulfonation is desirable in order to avoid having to separate unsulfonated components, a relatively high excess of sulfonating agent has to be employed in the sulfonation. As a result, the sulfonates, which are obtained as final products, are relatively dark-colored, and require bleaching with oxygen-yielding bleaches prior to their technical use. The time required for the bleaching process can be shortened or the amount of bleach that is necessary can be considerably reduced by carrying out the sulfonation of the olefins in two stages. However, a one-stage process is preferred if it does not entail any appreciable losses in the quality or quantity of product thereby obtained.

It is furthermore known in the prior art to carry out the sulfonation of olefins using as the sulfonation agent chloro-sulfonic acid, or sulfur trioxide, the sulfonation being effected in the presence of inert solvents such as a paraffinic hydrocarbon, chlorinated hydrocarbon or liquid sulfur dioxide. The results achieved have not been satisfactory, and it has, therefore, been proposed to use solvents which form stable addition products with sulfur trioxide as, for example, dioxane. If in the first stage of the process the sulfonating agent is dissolved in dioxane and the mixture is contacted in a second stage with the olefin to be sulfonated, relatively light-colored sulfonation products are obtained. Such a process, however, is complicated, and it is also encumbered by necessity for the recovery of the solvent, which recovery always entails losses and additional expense. The large amounts of liquid that have to be handled in a sulfonation effected according to this procedure also necessitate the installation of larger systems.

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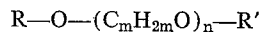
It is, therefore, an object of the present invention to provide a process of producing light-colored olefin sulfonates having a low content of non-surfactant components in a one-stage procedure and without the necessity for employing liquid solvents.

It is another object to provide a process of producing light-colored olefin sulfonates without resort to separate bleaching treatment of the sulfonation products.

It is an additional object to provide olefin sulfonates which are characterized by their relatively light color and by their low content of non-surfactants.

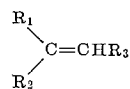
These and other objects will become apparent in the following description and claims.

The present invention is directed to a process for the preparation of surface-active olefin sulfonates by the sulfonation of olefins with gaseous sulfur trioxide diluted with air or an inert gas, followed by alkaline hydrolysis, wherein as initial material for the preparation of the technically valuable sulfonation products there are employed aliphatic monoolefins having 8 to 22 carbon atoms in their molecules and wherein the sulfonation is carried out in the presence of a polyglycol ether of the formula



wherein R represents alkyl having 8 to 22 carbon atoms, R' represents hydrogen or alkyl having 1 to 6 carbon atoms, *m* has a value of 2 or 3, and *n* represents a whole number of from 1 to 20.

The aliphatic monoolefins serving as starting materials preferably contain 12 to 18 carbon atoms and have either a middle or terminal double bond. The monoolefins can be straight-chain or branched-chain. Olefins having an unbranched chain, and olefins of the formula



are preferred. In the last formula, R₁ and R₂ represent straight-chain, alkyl radicals having 1 to 18 carbon atoms, R₃ represents hydrogen or a straight-chain alkyl radical having 1 to 18 carbon atoms, at least one of the alkyl radicals containing at least 6 carbon atoms. Mixtures of olefins of different structure and chain lengths can also be used. The starting olefins can be obtained in the known manner from hydrocarbon mixtures or by synthesis from olefins of low molecular weight. They do not need to be entirely pure, but may contain small amounts, though not more than 10%, of paraffins and/or diolefins.

Examples of suitable olefins are n-decene-1, n-undecene-1, n-tridecene-1, n-dodecene-1, n-tetra-decene-1, n-pentadecene-1, n-hexadecene-1, n-heptadecene-1, n-octadecene-1 and mixtures thereof, sec. n-decene, sec. n-undecene, sec. n-tridecene, sec. n-tetradecene, sec. n-pentadecene, sec. n-hexadecene, sec. n-heptadecene and sec. n-octadecene wherein the double bond is distributed over the chain.

The polyglycol ethers of the formula as set out above can be prepared by the conventional methods from primary or secondary alcohols by reaction thereof with ethylene oxide or propylene oxide. Mixtures of ethylene oxide and propylene oxide can also be used, or the two alkoxides can be used successively. The alkoxylation can be followed by an alkylation of the free hydroxyl groups which can be conducted according to the known and conventional methods. The two alkyl radicals R and R' can be straight- or branch-chained or, alternatively, can be cyclic. Ethoxylation products of straight-chain primary or secondary alcohols, which are preferred for use herein, have from 2 to 12 ethylene glycol ether groups as well as one free hydroxyl group, the alkyl group R having from 10 to 18 carbon atoms.

Examples of suitable polyglycol ethers are the di-, tri-, tetra-, penta-, hexa-, hepta-, octa-, nona-, deca-, undeca-, and dodeca-ethylene glycol ether of primary or secondary n-decanol, n-dodecanol, n-tetradecanol, n-hexadecanol and n-octadecanol. Said polyglycol ethers may be etherified with 1 to 10 propylene glycol ether groups or with methyl, ethyl, propyl or isopropyl groups.

The polyglycol ethers obtained by the addition of alkylene oxide onto long-chain alcohols may also contain unetherified alcohols. These do not give rise to any problems and may remain in the mixture. In the subsequent sulfonation reaction, they are converted into their corresponding alkyl sulfates.

The ratio of olefin to polyglycol ether in the mixture amounts advantageously to from 9:1 to 1:4 parts by weight. These ratios can be exceeded in either direction, but in that case, the color of the reaction products increasingly deteriorates.

The sulfonation is conducted according to the known method. If gaseous sulfur trioxide is used as the sulfonating agent, it is used diluted with air or with an inert gas, the sulfur trioxide concentration amounting to from 0.5 to 10 and preferably from 1 to 5 percent by volume. Illustrative of the inert gases, which can be employed as diluent, are, for example, nitrogen, carbon dioxide or sulfur dioxide.

Instead of the sulfur trioxide, as sulfonating agent, the equivalent amount of chlorosulfonic acid can also be advantageously used. The hydrogen chloride that forms in the reaction should be removed, for example, by passing a stream of air or inert gas through the reaction mixture, and continuing this operation for some time after the sulfonation has ended, or by alternatively operating under vacuum.

The sulfonation can be carried out discontinuously, or continuously, using a cocurrent or countercurrent flow. It is recommended, however, that the reactants be intensively mixed during the sulfonation reaction.

The sulfonation reaction takes place at temperatures ranging from 0° to 60° C., and preferably at temperatures of from 10° to 40° C. As the reaction is an exothermic one, additional heat input is generally unnecessary. In most cases it is necessary to remove the excess reaction heat, and, accordingly, it is recommended that the optimum temperature be maintained by appropriate cooling of the reaction vessel.

The reaction time depends to a great extent on the conditions selected for carrying out the reaction, such as the temperature, the concentration of the sulfonating agent and the type of reaction apparatus used. Short reaction times can be achieved particularly when the reaction mixture is intensively agitated by means of appropriate mechanical devices or is sprayed, or when reaction apparatus, which is used, are those operating on the thin layer or annular gap principle. If small quantities are used, the mixing action that is produced by the introduction of the inert gas stream into the reaction mixture will suffice under certain circumstances for achieving the desired degree of sulfonation and nature of sulfonation product.

The sulfonation is continued until at least 1.0 and no more than 1.3 mols, and preferably 1.05 to 1.2 mols of sulfur trioxide have been absorbed per mol of sulfonatable material. The olefins and the polyglycol ethers which contain a free hydroxyl group in their molecules are considered as the sulfonatable materials. With reference to these data, it is to be borne in mind that only the sulfur trioxide that remains in the product enters into the computation of the molar ratio.

The sulfonation can also be carried out in two stages. In this case it is preferred to proceed in a first stage wherein the sulfonation is carried out as described above, at a reaction temperature of 10 to 40° C. and with a sulfur trioxide concentration in the sulfonating gas of 1 to 5% by volume. After the mixture to be sulfonated has

absorbed 60 to 80% of the total amount of sulfur trioxide to be used, it is advantageous that in the second stage the temperature be reduced by about 5 to 10° C. and the sulfur trioxide concentration in the gas reduced by at least 20%, i.e., to a range of from 0.5 to 4% by volume. It may, furthermore, be advantageous to increase the rate of feed of the sulfonation gas in the second stage, or reduce the time of action of the sulfonating agent. Finally, it is also possible to use gaseous sulfur trioxide in the first sulfonating stage, as described above, and thereafter to use chlorosulfonic acid in the second stage. The amounts of sulfonating agent to be used per mol of sulfonatable material likewise amount to 1.0 to 1.3 mols, of which 0.5 to 1.0 and preferably 0.6 to 0.9 mol can be sulfur trioxide and 0.2 to 0.6 and preferably 0.3 to 0.5 mol can be chlorosulfonic acid. During the addition of the chlorosulfonic acid, it is advantageous to pass a strong stream of air or inert gas through the reaction mixture, whereby there is removed a considerable part of the hydrogen chloride formed in the reaction. The introduction of air or inert gas into the reaction mixture should be continued for some time after all of the chlorosulfonic acid has been added. In this manner, it is possible to obtain a sulfonation product which is substantially free of chlorine ions.

Often a sulfonation product having an even lighter color can be obtained by this two-stage sulfonation. In most cases, however, such a procedure is unnecessary.

The sulfonation products as obtained according to the described procedures are then neutralized with alkalis, ammonia, organic ammonium bases or alkaline earths and thereafter hydrolyzed by heating in an aqueous solution to temperatures of 80 to 200° C., using therefor that amount of water whereby the crude sulfonation product is in the form of a 7 to 75% solution. Hydrolysis at temperatures above 100° C. is carried out in pressure vessels. In general, 60 to 180 minutes are required for hydrolysis at temperatures of up to 100° C., and from 5 to 15 minutes are needed for hydrolysis at 200° C. The quantity of the alkaline reacting substance used for the neutralization is calculated so that, after neutralization of the sulfonic acid and any excess sulfonating agent that might be present, enough still remains for the neutralization of the sulfonic acid that is formed in the hydrolysis. It is preferable to work with an excess that can amount to as much as 20% of the theoretically necessary amount of alkali. Particularly suitable neutralization agents are the hydroxides, carbonates and bicarbonates of sodium, potassium and ammonium, as well as organic bases, such as the primary, secondary or tertiary amines or alkylolamines having 1 to 4 and preferably two carbon atoms per alkyl or alkylol radical. If organic bases are used, these react with the sultones contained in the raw sulfonation product to form surface-active alkylsulfobetaines. It is also possible however, first to neutralize the free sulfonic acids with an alkali metal or alkaline earth metal alkaline reacting agent and then to add one of the above-mentioned organic bases in such quantity that in the subsequent hydrolysis the sultones contained in the saponifiable portion are converted into alkylsulfobetaines.

If the hydrolysis of the raw sulfonation product is carried out at temperatures substantially higher than 100° C., a saponification of the polyglycol ether sulfates occurs which increases with the temperature and the heating time. However, since the unsulfated alkylpolyglycol ethers that develop also possess valuable surface-active properties, particularly when the number of ethylene glycol groups in the molecule is greater than 2, an additional possibility is thus provided for varying the composition of the products of the process and adapting them to a particular application.

If for any particular purpose a still light colored sulfonation product is desired, a bleaching treatment can be carried out with the conventional oxygen-yielding

bleaches. The sulfonation products obtained by the process according to the invention contain few, if any, colored by-products and are characterized by their excellent bleachability and low consumption of bleaching agent. The hydrolysis and the bleaching can be carried out simultaneously.

The light-colored mixtures manufactured according to the invention consist almost exclusively of surface-active materials. Depending on the procedure that is followed, there are recovered mixtures of olefin sulfonates, or mixtures thereof with alkylsulfobetaines and alkyl polyglycol ether sulfates, or mixtures thereof with unsulfated alkyl polyglycol ethers or dialkyl polyglycol ethers and fatty alcohol sulfates if the polyglycol ethers used as the starting substances contained free fatty alcohols. The unsulfonated olefin content of the mixtures, however, amounts to less than 5%.

The products of the process have outstanding cleaning, wetting, emulsifying and dispersing properties and can be used in admixture with other wash-active substances and conventional synthetic agents and additives in the formulation of detergents and washing compounds. Further, they can be used advantageously as additives in textile adjuvants. The properties of the products can be extensively adapted to a particular application. If the starting materials include polyglycol ethers having a free hydroxyl group and the hydrolysis is carried out at temperatures of about 100° C., relatively high-sudsing products are produced which have a high cleaning power, such as is desired in fine laundry detergents, foaming cleaners and bathing and hair washing detergents. The saponification of sulfated polyethylene glycol ethers or the use of dialkylated polyethylene glycol ethers results, if there are at least 4 to 5 glycol ether groups in the molecule, in low-sudsing preparations, suitable particularly for use as washing machine detergents. If the end products contain mainly dialkylated or unsulfated polypropylene glycol ethers or unsulfated glycol ethers having 1 to 3 ethylene glycol ether groups in their molecules, the products are suitable primarily as emulsifiers.

The fact that the sulfonation of the mixtures of olefins and polyglycol ethers even in the absence of inert solvents results in particularly light-colored sulfonated products is surprising, since both the olefins and the polyglycol ethers having a free hydroxyl group, if sulfonated separately with SO₃ under the same conditions, form relatively dark-colored sulfonation products which are technically usable only after intensive bleaching has been carried out. As, furthermore, mixtures of alkyl polyglycol ethers and other sulfonatable materials, especially alkylbenzenes or fatty acid esters, under the same conditions result in the production of dark-colored sulfonation products having a low degree of sulfonation, it was

not to be expected that the mixtures used according to the invention would behave in a fundamentally different manner.

The following examples will further illustrate how the said invention may be carried out in practice, but the invention is not restricted to these examples:

The olefins used in the following examples contained about 2% paraffinic hydrocarbons. The color values as recited were determined in a 4" cell in the Lovibond Tintometer employing aqueous solutions which contained 5 wt. percent of hydrolyzed and neutralized sulfonation product. The unsulfonated olefins present in the end product were determined by extracting a sample of the hydrolyzed product three to four times using the same volume of benzene having a boiling point of 80 to 110° C. each time, removing the extractant by distillation, and determining the iodine number of the remaining oil. The amount of olefin thereby computed was added to the paraffins that were introduced with the starting material. The value thus obtained is set out hereinafter as the "water-insoluble portion" in percent by weight of water-free end product. The mere weighing out of the benzene extract would produce false results since the polyglycol ethers are partially soluble in the organic phase.

In Example 13, the use of a two-stage procedure is exemplified; in the first stage, the sulfonating was carried out with sulfur trioxide, and in the second stage chlorosulfonic acid was employed as sulfonating agent.

EXAMPLE 1

In a flask with a 2-liter capacity, equipped with a thermometer, a gas introduction tube extending down to the bottom of the flask, a gas exhaust tube and a high-speed agitator, a mixture of 112 g. (0.5 mol) of hexadecene-1 (iodine No.=113) and 141 g. (0.5 mol) of a primary alcohol having a chain length of C₁₂ to C₁₄ that had been reacted with 2 mols of ethylene oxide were sulfonated, under intense agitation, by introducing into the olefin a gaseous mixture of SO₃ and air containing 3% of SO₂ by volume. 92 grams (1.15 moles) of SO₃ were introduced in the course of 45 minutes. The temperature of the reaction mixture was maintained at 20 to 35° C. by external cooling. After the sulfonation had been completed, the reaction product was poured with agitation into a solution of 52 g. (1.3 mols) of sodium hydroxide in 600 ml. of water and refluxed for 3 hours. The water-insoluble content of the reaction product was determined and found to amount to 3.3%. A 5% neutral aqueous solution gave the following color values: yellow=16, red=3.1, blue=0.

The examples listed in Table I were performed in like manner. In the table, the abbreviations EO and PO represent ethylene glycol ether groups and propylene glycol ether groups, respectively.

TABLE I

| Example | Olefin | Polyglycol Ether | Sulfonating Agent | Insoluble Portions, percent | Color Values | | |
|---------|-------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------|-------------------------------------------|-----------------------------|--------------|------|------|
| | | | | | Yellow | Red | Blue |
| 2..... | 112 g. (0.5 mol) of n-hexadecene with the double bond randomly distributed over the chain; I No.=111. | 141 g. (0.5 mol) prim. C ₁₂ -C ₁₄ alcohol + 2 EO. | 89 g. SO ₃ (1.11 mols)..... | 3.5 | 16 | 2.9 | 0 |
| 3..... | 98 g. (0.5 mol) of n-tetradecene with the double bond randomly distributed over the chain; I No.=130. | 141 g. (0.5 mol) same as Example 2. | 92 g. SO ₃ (1.15 mols)..... | 3.4 | 15 | 2.5 | 0 |
| 4..... | 116 g. (0.5 mol) of C ₁₅ -C ₁₈ alpha olefins, C No. 16.5, I No.=114. | 141 g. (0.5 mol) same as Example 2. | 92 g. SO ₃ (1.15 mols)..... | 3.7 | 14 | 2.1 | 0 |
| 5..... | 156 g. (0.67 mol) same as Example 4. | 93 g. (0.33 mol) same as Example 2. | 92 g. SO ₃ (1.15 mols)..... | 4.0 | 20 | 4.6 | 0 |
| 6..... | 174 g. (0.75 mol) same as Example 4. | 70.5 g. (0.25 mol) same as Example 2. | 92 g. SO ₃ (1.15 mols)..... | 4.4 | 25 | 6.5 | 0 |
| 7..... | 200 g. (0.9 mol) same as Example 4. | 28 g. (0.1 mol) same as Example 2. | 92 g. SO ₃ (1.15 mols)..... | 4.8 | 27 | 13.5 | 0 |
| 8..... | 174 g. (0.75 mol) same as Example 4. | 86 g. (0.25 mol) prim. C ₁₂ -C ₁₄ alcohol + 3.1 EO. | 92 g. SO ₃ (1.15 mols)..... | 3.6 | 15 | 3.4 | 0 |
| 9..... | 116 g. (0.5 mol) same as Example 4. | 171 g. (0.5 mol) same as Example 8. | 92 g. SO ₃ (1.15 mols)..... | 3.4 | 15 | 5 | 0 |
| 10..... | 116 g. (0.5 mol) same as Example 4. | 160 g. (0.5 mol) sec. C ₁₂ -C ₁₅ alcohol + 3.1 EO. | 92 g. SO ₃ (1.15 mols)..... | 5.5 | 11 | 2.8 | 0 |
| 11..... | 186 g. (0.5 mol) same as Example 4. | 122 g. (0.2 mol) prim. C ₁₄ alcohol + 9 EO. | 92 g. SO ₃ (1.15 mols)..... | 4.0 | 17 | 3.0 | 0 |
| 12..... | 116 g. (0.5 mol) same as Example 4. | 141 g. (0.5 mol) same as Example 2. | 140 g. ClSO ₃ H (1.2 mols).... | 4.8 | 18 | 3.5 | 0 |

TABLE I—Continued

| Example | Olefin | Polyglycol Ether | Sulfonating Agent | Insoluble Portions, percent | Color Values | | |
|---------|-----------------------------------------------------------------------------------------|---------------------------------------------------------------|----------------------------------------------------------------------|-----------------------------|--------------|-----|------|
| | | | | | Yellow | Red | Blue |
| 13..... | 116 g. (0.5 mol) same as Example 4. | 141 g. (0.5 mol) same as Example 2. | (a) 0.7 mol of SO ₃ , (b) 0.5 mol of ClSO ₃ H. | 3.8 | 16 | 2 | 0 |
| 14..... | 93.5 g. (0.4 mol) C ₁₅ -C ₁₈ alpha olefins C No. 16.5; I No.=111. | 138 g. (0.2 mol) sec. C ₁₄ alcohol + 9 EO. | 56 g. SO ₃ (0.69 mol)..... | 4.2 | 7 | 1.2 | 0 |
| 15..... | 174 g. (0.75 mol) same as Example 14. | 173 g. (0.25 mol) same as Example 14. | 92 g. SO ₃ (1.15 mols)..... | 4.9 | 14 | 2.4 | 0 |
| 16..... | 209 g. (0.9 mol) same as Example 14. | 69.5 g. (0.1 mol) same as Example 14. | 92 g. SO ₃ (1.15 mols)..... | 3.8 | 18 | 4.7 | 0 |
| 17..... | 174 g. (0.75 mol) same as Example 14. | 189 g. (0.25 mol) prim. C ₁₂ alcohol + 11 EO. | 92 g. SO ₃ (1.15 mols)..... | 4.6 | 9 | 1.7 | 0 |
| 18..... | 193 g. (0.83 mol) same as Example 14. | 128 g. (0.17 mol) same as Example 17. | 92 g. SO ₃ (1.15 mols)..... | 4.2 | 9 | 1.9 | 0 |
| 19..... | 209 g. (0.9 mol) same as Example 14. | 75.5 g. (0.1 mol) same as Example 17. | 92 g. SO ₃ (1.15 mols)..... | 4.8 | 16 | 3.8 | 0 |
| 20..... | 172 g. (0.8 mol) 2-hexyldecene-1 I No.=121. | 120 g. (0.2 mol) prim. C ₁₄ alcohol + 9 EO. | 96 g. SO ₃ (1.2 mols)..... | 3.3 | 8 | 1.0 | 0 |
| 21..... | 172 g. (0.8 mol) 7-methylpenta-decene-6 I No.=120. | 125 g. (0.2 mol) prim. C ₁₂ alcohol + 6 EO + 3 PO. | 88 g. SO ₃ (1.1 mols)..... | 3.8 | 11 | 2.1 | 0 |

To demonstrate the technical effect, a series of comparative tests was carried out under comparative conditions. The results of the tests are summarized in Table II. In Experiments A and B, the olefins used in Examples 2 and 4 were sulfonated separately, and in Experiment C, the alkylpolyglycol ether used in Examples 1 to 4 was sulfonated separately. The products contain a higher percentage of unsulfonated, water-insoluble components and are dark colored. Experiments D and E show that mixtures of polyglycol ethers and other sulfonatable starting materials are substantially less effective than the mixtures which are to be processed according to the invention.

20 least one of the alkyl substituents contains at least 6 carbon atoms.

4. A process according to claim 1 wherein gaseous sulfur trioxide diluted with air or inert gas is employed as the sulfonating agent, the sulfur trioxide concentration amounting to from 0.5 to 10% by volume.

5. A process according to claim 4 wherein the sulfur trioxide concentration amounts to from 1 to 5% by volume.

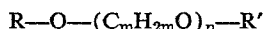
6. A process according to claim 1 wherein said hydrolysis is effected by adding water and an alkaline reacting agent to said sulfonation reaction product and heating the

TABLE II

| Experiment | Starting Material | SO ₃ | Conditions of Reaction | Insoluble Portions, Percent | Color Values | | |
|------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------|-------------------------------------------------------------------------------------------------|-----------------------------|---------------------------------|------|------|
| | | | | | Yellow | Red | Blue |
| A..... | 232 g. (1.0 mol) C ₁₅ -C ₁₈ olefin, Average C No. 16.5; I No.=114. | 92 g. (1.15 mols)..... | Same as Example 1..... | 6.3 | >27 | >27 | 3.3 |
| B..... | 224 g. (1.0 mol) n-hexadecene with double bond randomly distributed over the chain; I No.=111. | 92 g. (1.15 mols)..... | do..... | 5.6 | 26 | 23 | 2.8 |
| C..... | 282 g. (1 mol) prim. C ₁₂ -C ₁₄ alcohol plus 2 EO; hydroxyl No.=199. | 92 g. (1.15 mols)..... | do..... | 4.8 | 26 | 11.4 | 0 |
| D..... | 121 g. (0.5 mol) dodecylbenzene plus 141 g. (0.5 mol) primary C ₁₂ -C ₁₄ alcohol plus 2 EO. | 92 g. (1.15 mols)..... | ¼ hr. at 60-65° C. 10 minutes post-reaction, 5 vol.-Percent mixture of SO ₃ and air. | 29.0 | >27 | 18 | 4 |
| E..... | 118 g. (0.5 mol) hydrogenated palm kernel fatty acid methyl ester (C ₁₂ -C ₁₈) plus 141 g. (0.5 mol) prim. C ₁₂ -C ₁₄ alcohol plus 2 EO. | 96 g. (1.2 mols)..... | ¼ hr. at 80° C. 10 minutes post-reaction, 5 vol.-Percent mixture of SO ₃ and air. | 46.0 | Not measurable because too dark | | |

We claim:

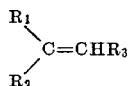
1. A process for the preparation of surface-active sulfonates which comprises reacting an olefin having 8 to 22 carbon atoms in its molecule with 1.0 to 1.3 mols per mol of said olefin gaseous sulfur trioxide diluted with an inert gas as sulfonating agent, in the presence of a polyglycol ether having the formula:



wherein R represents alkyl of 8 to 22 carbon atoms, R' represents a member selected from the group of hydrogen and alkyl having 1 to 6 carbon atoms, m is 2 or 3 and n is a whole number from 1 to 20 at a temperature of from 0 to 60° C., hydrolyzing the sulfonated olefin product thereby produced and thereafter neutralizing the resulting mixture with an alkaline neutralizing agent; the ratio of said olefin to said polyglycol ether is from 9:1 to 1:4 parts by weight.

2. A process according to claim 1 wherein said olefin is straight chained, has an internal or terminal double bond and contains from 12 to 18 carbon atoms.

3. A process according to claim 1 wherein said olefin has the formula



wherein R₁ and R₂ are each straight-chain alkyl having 1 to 18 carbon atoms and R₃ is a member selected from the group consisting of hydrogen and straight-chain alkyl having 1 to 18 carbon atoms, with the proviso that at

45 resultant mixture to a temperature of from 80 to 200° C.

7. A process according to claim 6 wherein sufficient water is added to the sulfonation reaction product to form a 7 to 75% solution.

8. A process according to claim 6 wherein said alkaline reacting agent is employed in an excess amounting to up to 20% of that theoretically necessary.

9. The surface-active olefin sulfonate characterized by light color produced by the process of claim 1.

10. The surface-active olefin sulfonate characterized by light color produced by the process of claim 6.

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