

1

3,240,819

ETHENOXY-SUBSTITUTED ALKANOLS

Van R. Gaertner and Donald N. Van Eenam, Dayton, Ohio, assignors to Monsanto Company, a corporation of Delaware

No Drawing. Filed Feb. 17, 1960, Ser. No. 9,197

2 Claims. (Cl. 260-615)

This invention relates to ethenoxy-substituted alkanols, including chloro and alkoxy derivatives thereof. In one aspect, this invention relates to ethenoxy-substituted alkanols as new compounds. In another aspect, this invention relates to methods for preparing ethenoxy-substituted alkanols from a 1,2-epoxyalkane and an ethylene glycol. In another aspect, this invention relates to surfactant compositions which have very high deterative properties as well as good wetting and lime soap dispersion efficiencies. In another aspect, this invention relates to methods for improving the deterative, wetting and lime soap dispersion efficiencies of detergent and soap compositions.

It is well known that many of the present soap and detergent compositions contain materials which serve to increase the wetting, deterative and lime soap dispersion efficiencies of the composition. Some of these materials are very effective in performing their intended purpose, whereas others of these materials are not as effective as desired. It is very desirable to find a surfactant having high deterative properties because less of the surfactant is required in a detergent or soap composition for manufacturing a composition of a predetermined deterative rating. Also, a detergent or soap composition containing a smaller proportion of the active ingredient is generally more economical to manufacture. Further, detergent compositions containing a smaller proportion of active ingredient are usually more readily processed in that fewer problems are encountered in the spray drying and sizing steps. Consequently, it is desirable to find new surface active materials having good surface active properties, particularly high deterative efficiencies, for use in detergent and soap compositions.

An object of this invention is to provide ethenoxy-substituted alkanols as new compounds.

Another object of this invention is to provide methods for preparing the ethenoxy-substituted alkanols from an epoxy-alkane and an ethylene glycol.

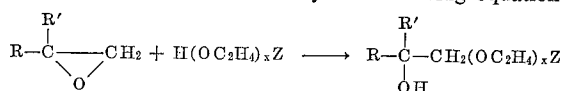
Another object of this invention is to provide surfactant compositions which have improved surface active properties, particularly high deterative efficiencies.

Another object of this invention is to provide new all-purpose soap compositions which have improved deterative, wetting, and lime soap dispersion efficiencies.

Another object of this invention is to provide a method for increasing the deterative, wetting, and lime soap dispersion efficiencies of soap and detergent compositions.

Other aspects, objects and advantages of this invention will be apparent from a consideration of the accompanying disclosure and appended claims.

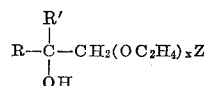
In accordance with this invention, an epoxyalkane is reacted with an ethylene glycol to form an ethenoxy-substituted alkanol as illustrated by the following equation:



wherein R and R' are selected from the group consisting of hydrogen and alkyl radicals, said R and R' being the same or different, said R and R' together having a total of from 6 to 24 carbon atoms, x is an integer of from 1 to 100, and Z is selected from the group consisting of chloro, hydroxyl, and alkoxy radicals.

2

Further, in accordance with this invention, there are provided, as new compounds, ethenoxy-substituted alkanols of the formula



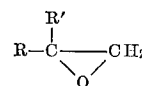
wherein R, R', x and Z are as above defined.

Further, in accordance with the present invention, there are provided new surfactant compositions comprising, as the active ingredient, an ethenoxy-substituted alkanol of the formula given above.

Further, in accordance with this invention, there are provided new all-purpose detergent compositions, comprising a sodium, potassium or ammonium salt of the long-chain fatty acids and, as an essential ingredient, an ethenoxy-substituted alkanol of the formula given above.

Further, in accordance with this invention, there are provided methods for increasing the deterative, wetting, and lime soap dispersion efficiencies of soap-containing detergent compositions by adding an ethenoxy-substituted alkanol of the formula given above to a sodium, potassium, or ammonium long-chain fatty acid soap.

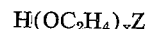
The epoxyalkane reactants useful for the preparation of the presently provided new compounds of our invention can be any epoxyalkane having a terminal group; i.e., an epoxyalkane having the structure



wherein R and R' are selected from the group consisting of hydrogen and alkyl radicals, said R and R' being the same or different, said R and R' together having a total of from 6 to 24 carbon atoms. The carbon atoms of the alkyl radicals may be arranged in either a straight-chain or a branched-chain configuration. Illustrative examples of some suitable alkyl radicals include the methyl, ethyl, butyl, isohexyl, 2-ethylhexyl, isononyl, n-dodecyl, tert-dodecyl, 2-propylheptyl, 5-ethylnonyl, 2-butyloctyl, n-tetradecyl, n-pentadecyl, tert-octadecyl, 2,6,8-trimethylnonyl, and 7-ethyl-2-methyl-4-undecyl radicals. The alkyl radicals may also include unsaturated alkyl radicals such as oleyl, dodecenyl, hexadecenyl, and the like. An especially valuable class of epoxides is derived from epoxidation of olefins formed either by polymerization of olefin monomers or by dehydration of the alcohols derived from an olefin monomer, dimer, trimer, tetramer, pentamer, or the like, carbon monoxide and hydrogen by the "Oxo" process.

Illustrative examples of some epoxyalkanes which can be used as reactants in the process of this invention are as follows: 1,2-epoxyoctane, 1,2-epoxydecane, 1,2-epoxydodecane, 2-butyl-1,2-epoxyoctane, 1,2-epoxytetradecane, 7-ethyl-2-methyl-1,2-epoxyundecane, 2,6,8-trimethyl-1,2-epoxynonane, 1,2-epoxyhexadecane, 1,2-epoxyoctadecane, 1,2-epoxyeicosane, 2,4-dimethyl-1,2-epoxydodecane, and 1,2-epoxytetracosane.

The ethylene glycol reactants which are useful for the preparation of the presently provided new compounds of our invention can be represented by the formula



wherein x and Z are as above defined. In this formula, Z is chloro, hydroxyl or a lower alkoxy radical, preferably an alkoxy radical wherein the alkyl group contains from 1 to 6 carbon atoms, such as methyl, ethyl, isobutyl, hexyl, and the like. In this formula, x is an integer of

from 1 to 100, inclusive, and represents the number of ethenoxy groups in the ethylene glycol molecule. Thus, in this specification and claims, the term "ethylene glycol" is considered to include not only the compound ethylene glycol where x is 1, but also includes the polyethylene glycols where x is greater than 1. Although x can be an integer which is a whole number, usually x is not a whole number except in the case of ethylene glycol because the polyethylene glycols usually comprise a mixture of polymers having various numbers of ethenoxy groups. Customarily, the polyethylene glycols are identified by the average molecular weight of the product and a polyethylene glycol of average molecular weight, say, 200, will contain some polyethylene glycol molecules of substantially less than 200 molecular weight as well as some molecules of substantially greater than 200 molecular weight. For this reason, the new compounds are described in this specification and claims with a recitation of the average molecular weight of the polyethylene glycol from which the product is made instead of being described as compounds of specific compositions. However, a polyethylene glycol of average molecular weight 200 can be identified as a tetraethylene glycol since there are approximately 4.1 ethenoxy groups in the molecule and x in the formula above is 4.1. Similarly, a polyethylene glycol of average molecular weight 300 can be identified as heptaethylene glycol since there are 6.4 ethenoxy groups per molecule and x is 6.4. In the same manner, other polyethylene glycols of defined average molecular weight can be assigned generic names such as nonaethylene glycol where the average molecular weight is 400, tridecaethylene glycol where the average molecular weight is 600, heptadecaethylene glycol where the average molecular weight is 800, docosaethylene glycol where the average molecular weight is 1000, and the like.

The ethylene glycols, including ethylene glycol and polyethylene glycol and the chloro-substituted and alkoxyl-substituted derivatives thereof, are readily available commercial products. Well known polyethylene glycols are available on the market under the trade name "Carbowax," manufactured by the Union Carbide Chemical Co. of New York. Various "Carbowax" polyethylene glycols are distinguished from each other by numbers which are indicative of the average molecular weight of the products; that is, Carbowax 200 polyethylene glycol has an average molecular weight of 190-210 and Carbowax 600 polyethylene glycol has an average molecular weight of 570-630. Although these commercially available polyethylene glycols are preferred in this invention, any available polyethylene glycols can be used in the process of this invention.

Reaction of the epoxyalkane and the ethylene glycol takes place readily by contacting the reactants in either an alkaline or an acidic reaction system, advantageously at an elevated temperature, and then allowing the resulting reaction mixture to stand until the desired product has been formed. The reaction will not take place in neutral reaction systems; either an acidic or an alkaline material, which can be present in catalytic amounts, must be present in the reaction mixture. The acidic type catalyst can be either hydrofluoric acid, perchloric acid, alkanesulfonic acid, arenesulfonic acid, or a Lewis type acid, such as aluminum chloride, boron trifluoride, stannic chloride, ferric chloride, and the like. Suitable alkaline type catalysts include the alkaline earth metals or the alkali metals, oxides, or hydroxides thereof. For example, the alkaline earth metals can be calcium, magnesium, barium, strontium, or the like whereas the alkali metals can be sodium, potassium, or lithium. Preferably, the catalyst is an acidic one and boron trifluoride is the preferred catalyst of this type. The amount of catalyst present in the reaction zone can be varied over wide limits as determined by the particular reactants used, by the temperature desired, and by the reaction time se-

lected. Ordinarily, the amount of catalyst will be between about 0.01% and 5.0% by weight of the amount of ethylene glycol reactant present.

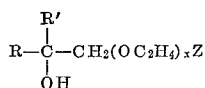
The reaction temperature used in the process of this invention depends to some extent upon the type of catalyst present in the reaction zone. Thus, some reaction takes place at a temperature slightly above room temperature when using an acidic type catalyst whereas the temperature must usually be above 50° C. when an alkaline type catalyst is used. Preferably, the temperature is maintained in the range of from 50° C. to 100° C. using an acidic type catalyst and between 80° C. and 160° C. when using an alkaline type catalyst. Temperatures below these preferred temperature levels can be used; however, the reaction times are rather long and it is desirable to use temperatures above these lower limits in order to obtain a good yield of the product in a suitable reaction time. Temperatures above the specified upper limits should not be used since the reactants are unstable at the more elevated temperature; that is, the ethylene glycols are decomposed to eliminate water at temperatures above 160° C.

The reaction of this invention is usually carried out at substantially atmospheric pressure although either sub-atmospheric or superatmospheric pressure can be used if desired.

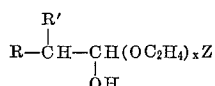
Although the epoxyalkane and ethylene glycol reactants can be reacted together in approximately stoichiometric proportions, it is usually preferred to have an excess of the ethylene glycol reactant present in the system at all times in order to avoid the formation of undesirable side-reaction products. Preferably, the ethylene glycol reactant is present in an amount greater than 2 moles of ethylene glycol per mole of the epoxyalkane and in many reaction systems the proportions are substantially greater. In order to have an excess of the ethylene glycol reactant present at all times, the reaction is usually conducted by the slow or dropwise addition of the epoxyalkane to a mixture of the catalyst and the ethylene glycol.

The reaction of the epoxyalkane with the ethylene glycol is primarily an addition-type reaction resulting in the formation of a single product. However, some reaction conditions may result in the formation of by-products, necessitating a separation step to remove the same. These side-reaction products can usually be removed by either vacuum steam distillation of the reaction mixture at an elevated temperature or solvent extraction of the reaction product with an immiscible solvent such as hexane. In the removal of the side-reaction products, the presence of an excess of the ethylene glycol reactant improves the purification step since it aids in keeping the ethenoxy-substituted alkanol in solution in the ethylene glycol. After removal of the side-reaction products, the excess ethylene glycol is removed from the reaction product by extraction with a hot salt solution in which the ethylene glycol is soluble and the ethenoxy-substituted alkanol is substantially insoluble. Suitable salt solutions include aqueous solutions of sodium chloride, potassium chloride and the like. The salt is present in the solution in an amount which gives a saturated solution at the elevated temperature where the extraction is carried out. This separation step cannot be carried out at room temperature but must be carried out at an elevated temperature since the ethenoxy-substituted alkanols of this invention are more soluble in cold water than in hot water. Preferably, the extraction is carried out at a temperature near the boiling point of the salt solution and in all cases at a temperature above 60° C. After removal of the unconverted ethylene glycol reactant, the product is dried to remove any water which may be present. The drying can be accomplished either by heating the product slightly under a reduced pressure or by contacting the product with a drying agent such as magnesium sulfate or calcium sulfate.

The novel ethenoxy-substituted alkanols of this invention have the structural formula



wherein R, R', x, and Z are as above defined. Ordinarily, the major product of the process of this invention has the structure shown with the hydroxyl group attached to the carbon atom in the 2-position with respect to the oxygen atom of the ethenoxy group. However, this reaction also results in the formation of some polyethenoxy-substituted alkanols of the structure



wherein the hydroxyl group is attached to the carbon atom in the 1-position. Since the predominant product is one in which the hydroxyl group is attached to the carbon atom in the 2-position, it is intended in this specification and claims that the formula having the hydroxyl group attached to the carbon atom in the 2-position be considered to also cover the compound wherein the hydroxyl methyl group is attached to the carbon atom in the 1-position.

The novel compound of this invention is a 1-[(2-hydroxyethoxy)polyethenoxy]-2-alkanol or a 1-[(2-hydroxyethoxy)-ethenoxy]-2-alkanol where Z is hydroxyl, a 1-[(2-chloroethoxy)-polyethenoxy]-2-alkanol or a 1-[(2-chloroethoxy)ethenoxy]-2-alkanol where Z is chloro, or a 1-[(2-alkoxyethoxy)polyethenoxy]-2-alkanol or a 1-[(2-alkoxyethoxy)ethenoxy]-2-alkanol where Z is alkoxy. Illustrative examples of some of these novel compounds are as follows:

1-[(2-hydroxyethoxy)ethenoxy]-2-octadecanol
1-[(2-hydroxyethoxy)polyethenoxy]-2-decanol
1-[(2-hydroxyethoxy)polyethenoxy]-2-hexadecanol
1-[(2-hydroxyethoxy)polyethenoxy]-2-octadecanol
1-[(2-hydroxyethoxy)polyethenoxy]-2-tetracosanol
1-[(2-hydroxyethoxy)polyethenoxy]-4-butyl-2-dodecanol
1-[(2-chloroethoxy)polyethenoxy]-2-octanol
1-[(2-chloroethoxy)polyethenoxy]-2-heptadecanol
1-[(2-chloroethoxy)polyethenoxy]-3,4-dimethyl-2-dodecanol
1-[(2-methoxyethoxy)polyethenoxy]-2-dodecanol
1-[(2-ethoxyethoxy)polyethenoxy]-2-octadecanol
1-[(2-butoxyethoxy)polyethenoxy]-2-decanol
1-[(2-propoxyethoxy)polyethenoxy]-7-ethyl-2-methyl-2-undecanol

The ethenoxy-substituted alkanols of this invention are stable, usually water soluble, waxy solids or viscous liquids which vary in color from colorless to light yellow. In general, they are more soluble in cold water than in hot water. They have cloud points which vary in range from approximately 0° C. to 100° C., depending upon the number of ethenoxy groups present in the molecule. These ethenoxy-substituted alkanols are valuable articles of commercial interest and have many varied uses, particularly as surfactants. They can be used as wetting, frothing, or washing agents in the treatment and processing of textiles, for dyeing, for pasting of dyestuffs, fulling, sizing, impregnating and bleaching and the like. In addition, these compounds are useful for preparing foam in fire extinguishers, for use as froth flotation agents, as air entering agents for concrete and cement, and as aids in the preparation of other articles of commerce. These compounds are particularly useful in soap and synthetic detergent compositions because many of these compounds have very high deterse efficiencies as well as good wetting and lime soap dispersion properties.

The advantages, desirability and usefulness of the present invention are illustrated by the following examples.

Example 1

In this example 36.9 g. (0.20 mole) of 1,2-epoxydodecane were reacted with 200 g. (0.50 mole) of a polyethylene glycol having an average molecular weight of 400. The polyethylene glycol and 0.5 ml. of a boron trifluoride-diethyl ether complex were placed in a reaction flask and heated to a temperature of 85° C. The 1,2-epoxydodecane was then added dropwise with stirring during a period of 3 hours to the reaction flask which was maintained at 85° C. After completing the addition of the 1,2-epoxydodecane, the reaction mixture was heated for an additional 30 minutes, with stirring, at the same temperature. At the end of this time, the reaction mixture was cooled to room temperature and extracted 3 times with 250 ml. portions of petroleum ether to remove oily side-reaction products. The reaction mixture was then extracted 3 times with a saturated sodium chloride solution at a temperature of 90° C. to remove unconverted polyethylene glycol. The product was then taken up in diethyl ether and dried with magnesium sulfate. After removal of the magnesium sulfate and added activated charcoal by filtration, the filtrate was heated in vacuum at a temperature up to 120° C. and a pressure of 15 mm. to remove the solvent and leave 75.6 g. of 1-[(2-hydroxyethoxy)polyethenoxy]-2-dodecanol prepared from a polyethylene glycol of 400 average molecular weight. This product was a colorless liquid having a refractive index n_D^{25} 1.4613 and a cloud point of 90° C. Analysis of this product was found to be 60.16 wt. percent carbon and 10.38 wt. percent hydrogen as compared with calculated values of 60.17 wt. percent carbon and 10.44 wt. percent hydrogen.

Example 2

In this example, 46.1 g. (0.25 mole) of 1,2-epoxydodecane were reacted with 180 g. (0.6 mole) of a polyethylene glycol having an average molecular weight of 300. The polyethylene glycol and 0.5 ml. of a boron trifluoride-diethyl ether complex were placed in a reaction flask and heated to a temperature of 80° C. The 1,2-epoxydodecane was then added dropwise with stirring during a period of 3.5 hours while maintaining the temperature between 80–85° C. After the addition of the 1,2-epoxydodecane was completed, the reaction mixture was heated for an additional 30 minutes at the same temperature. The reaction mixture was then cooled to room temperature and extracted 3 times with 250 ml. portions of petroleum ether to remove the oily side-reaction products. The reaction mixture was then extracted 3 times with saturated sodium chloride at a temperature of 90° C. to remove the unconverted polyethylene glycol. The product was then taken up in diethyl ether, dried with magnesium sulfate, and filtered to remove the magnesium sulfate and added activated charcoal. The filtrate obtained was evaporated under vacuum at a temperature of 120° C. and a pressure of 15 mm. to remove the diethyl ether and leave 76.5 g. of 1-[(2-hydroxyethoxy)polyethenoxy]-2-dodecanol prepared from a polyethylene glycol of 300 average molecular weight. This product was a colorless liquid having a refractive index n_D^{25} 1.4586 and a cloud point of 84° C.

Example 3

In this example, a polyethylene glycol having an average molecular weight of 600 was reacted with an epoxyalkane mixture containing 60% 1,2-epoxyhexadecane and 40% 1,2-epoxyoctadecane. The polyethylene glycol was placed in a reaction flask with 0.5 ml. of a boron trifluoride-diethyl ether complex and heated to a temperature of 85° C. Then the epoxyalkane was added dropwise with stirring during a period of 1.5 hours while maintaining the temperature at 85–90° C. After completion of the addition of the epoxyalkane, the reaction mixture was heated for an additional 0.5 hour at the same tempera-

ture. The reaction mixture was then vacuum steam distilled at a temperature up to 130° C. and a pressure of 15–25 mm. to remove oily side-reaction products. The mixture was then extracted 3 times with hot, saturated sodium chloride solution to remove unconverted polyethylene glycol. The product was then taken up in diethyl ether, dried over magnesium sulfate, and filtered to remove the magnesium sulfate and added activated charcoal. The filtrate was then heated under vacuum to obtain 75.6 g. of 1-[(2-hydroxyethoxy)-polyethenoxy]-2-*alkanol* prepared from a polyethylene glycol of 600 average molecular weight, wherein *alkanol* is a mixture of hexadecanol and octadecanol. The product was a straw-colored liquid having a refractive index n_D^{25} 1.4629 and a cloud point of about 100° C. The product solidified upon standing at room temperature.

Example 4

In this example, 150 g. (0.375 mole) of a polyethylene glycol having an average molecular weight of 400 was reacted with 37.8 g. (0.15 mole) of the 1,2-epoxyhexadecane and 1,2-epoxyoctadecane mixture used in Example 3. The polyethylene glycol and the epoxyalkane were reacted under the same conditions as in Example 3 using 0.5 ml. of a boron trifluoride-diethyl ether catalyst. After separation and purification of the reaction product in the same manner as in Example 3, there was obtained 77.7 g. of 1-[(2-hydroxyethoxy)polyethenoxy]-2-*alkanol* prepared for polyethylene glycol of 400 average molecular weight, wherein *alkanol* is a mixture of hexadecanol and octadecanol. The product which was obtained in 79.5% yield, was an off-white colored liquid having a refractive index n_D^{25} 1.4614 and a cloud point of 67° C.

Example 5

In this example, 150 g. (0.5 mole) of a polyethylene glycol having an average molecular weight of 300 was reacted with the epoxyalkane mixture of Example 3 under the conditions of Example 3 using 0.4 ml. of a boron trifluoride-diethyl ether complex or catalyst. After separation and purification of the reaction product according to Example 3, there was obtained 90.4 g. of 1-[(2-hydroxyethoxy)polyethenoxy]-2-*alkanol* prepared from a polyethylene glycol of 300 average molecular weight, wherein said *alkanol* is a mixture of hexadecanol and octadecanol. The product which was obtained in 82% yield, was a colorless liquid having a refractive index n_D^{25} 1.4599 and a cloud point of 40° C. Analysis of this product was 66.69 wt. percent carbon and 11.61 wt. percent hydrogen as compared with calculated values of 65.99 wt. percent carbon and 11.43 wt. percent hydrogen.

Example 6

In this example, a polyethylene glycol terminated with chlorine and having an average molecular weight of 610 was reacted with an epoxyalkane mixture containing 60% 1,2-epoxyhexadecane and a 40% 1,2-epoxyoctadecane. In a reaction flask were placed 244 g. (0.40 mole) of the polyethylene glycol chloride and 0.5 ml. of a boron trifluoride-diethyl ether complex. After heating the reaction flask and contents to a temperature of 50° C., 37.7 g. (0.15 mole) of the epoxyalkane was added dropwise with stirring during a period of two hours while maintaining the temperature at 50–55° C. Thereafter, the reaction mixture was heated for an additional 1 hour at a temperature in the range of 55–60° C. At the end of this time, the reaction mixture was cooled to room temperature and extracted three times with 250 ml. portions of petroleum ether followed by extraction with 3 portions of hot, 13% sodium chloride solution. The product was then taken up in a dichloromethane-diethyl ether mixture and dried over magnesium sulfate. The magnesium sulfate and added activated charcoal were removed by filtration. The product was heated under vacuum to remove the solvent mixture and leave 124.7 g.

(96.5% yield) of 1-[(2-chloroethoxy)polyethenoxy]-2-*alkanol* prepared from a polyethylene glycol chloride of 610 average molecular weight, wherein *alkanol* is a mixture of hexadecanol and octadecanol. The product was a colorless liquid having a cloud point of 69.5° C. and a freezing point of 28–29° C.

Example 7

In this example, a polyethylene glycol terminated with a methoxyl group and having an average molecular weight of 350 was reacted with an epoxyalkane mixture containing 60% 1,2-epoxyhexadecane and 40% 1,2-epoxyoctadecane. Into a reaction flask were placed 140 g. (0.40 mole) of the methoxypolyethylene glycol and 0.5 ml. of a boron trifluoride-diethyl ether complex. After heating the reaction flask and contents to a temperature of 80° C., 37.7 g. (0.15 mole) of the epoxyalkane was added dropwise with stirring during a period of 1.5 hours while maintaining the temperature at 78–85° C. Thereafter, the reaction mixture was heated for an additional 0.5 hour at the same temperature. At the end of this time, the reaction mixture was vacuum steam distilled at a temperature up to 150° C. and a pressure of 25–40 mm. to remove oily side-reaction products. The mixture was then extracted 3 times with hot, saturated sodium chloride solution to remove unconverted methoxypolyethylene glycol. The product was then taken up in diethyl ether, dried over magnesium sulfate, and filtered to remove magnesium sulfate and added activated charcoal. The filtrate was then heated under vacuum to obtain 86.1 g. (95.4% yield) of 1-[(2-methoxyethoxy)polyethenoxy]-2-*alkanol* prepared from a methoxypolyethylene glycol of 350 average molecular weight, wherein *alkanol* is a mixture of hexadecanol and octadecanol. The product was a colorless liquid having a cloud point of 50° C. and a freezing point of approximately 18° C.

Example 8

The wetting efficiencies of the ethenoxy-substituted alkanols of Examples 1 and 2 were determined by the Draves Wetting Test of the American Association of Textile Chemists. The following wetting times were measured at the concentrations shown:

Compound	Time in Seconds				
	0.5%	0.25%	0.125%	0.062%	0.031%
Compound of Ex. 1-----	10.5	15.0	30.8	109.4	+180
Compound of Ex. 2-----	3.6	7.4	15.3	30.1	+180

Example 9

The detergency properties of a number of the ethenoxy-substituted alkanols of this invention were measured by employing the method described by J. C. Harris and E. L. Brown in the Journal of American Oil Chemists Society, 27, 135–143 (1950). In this method, the detergency of the candidate compound was compared with the detergency of Gardinol WA, a commercial detergent produced by sulfating a mixture of alcohols, principally C₁₂ obtained by hydrogenating coconut oil fatty acids. The following detergency efficiencies were measured:

	0 p.p.m. Water Hard- ness 25° C.	50 p.p.m. Water Hard- ness 60° C.	300 p.p.m. Water Hard- ness 60° C.
Compound of Ex. 1-----	95	88	-----
Compound of Ex. 2-----	96	101	-----
Compound of Ex. 3-----	95	95	90
Compound of Ex. 4-----	102	112	104
Compound of Ex. 5-----	118	126	117
Compound of Ex. 6-----	-----	140	-----
Compound of Ex. 7-----	-----	139	-----

Using the detergency evaluation procedure noted above, the detergency of "built" materials of a number of the

ethenoxy-substituted alkanols of this invention were determined. The products were formulated by using 15% of the active surfactant with the balance of the formulation being composed of sodium tripolyphosphate, sodium tetrapyrophosphate, sodium silicate, and soda ash. The following results were obtained:

Compound	50 p.p.m. Water Hardness 60° C.	300 p.p.m. Water Hardness 60° C.
Compound of Ex. 1-----	101	-----
Compound of Ex. 2-----	106	-----
Compound of Ex. 3-----	102	107
Compound of Ex. 4-----	126	117
Compound of Ex. 5-----	135	121
Compound of Ex. 6-----	159	152
Compound of Ex. 7-----	150	166

Example 10

In this example, the lime soap dispersion efficiencies of the ethenoxy-substituted alkanols of Examples 3-7, were determined using the procedure described by J. C. Harris in ASTM Bulletin 140, p. 113, May, 1946. These results are reported in the table below wherein the dispersion number is equal to 10 times the milliliters of the test compound required to disperse 45.5 milligrams of calcium oleate formed.

Compound	Dispersion Number
Compound of Ex. 3-----	60
Compound of Ex. 4-----	40
Compound of Ex. 5-----	20
Compound of Ex. 6-----	60
Compound of Ex. 7-----	60

As surface active compositions, the ethenoxy-substituted alkanols of this invention comprise either the pure compounds or an admixture of the pure compounds with an adjuvant or diluent. Ordinarily, the compounds of this invention are employed in surface active applications in a dilute form where the adduct is dissolved or suspended in some liquid medium such as water or a hydrocarbon solvent. The ethenoxy-substituted alkanols of this invention can also be admixed with adjuvant materials, particularly when used in soap and synthetic detergent compositions, such as inorganic builders of the type such as carbonates, phosphates, silicates and fillers.

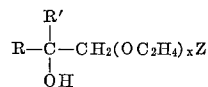
The new ethenoxy-substituted alkanols of this invention are particularly useful in soap and synthetic detergent compositions because these compounds possess good wetting, deterative and lime soap dispersion properties. The relative proportions of the compounds of this invention and the soap and/or synthetic detergent compositions may vary greatly depending upon the use intended. Although useful detergent compositions can be formed by mixing small proportions of soap with large proportions of the ethenoxy-substituted alkanols, usually the greatest value of soap compositions of the present invention lie in compositions having less than 70% by weight of the ethenoxy-

substituted alkanol. In general, it is preferred to incorporate into the soap composition about 5-50% by weight of the compound based on the total weight of the soap and the compound. Of course, other materials such as perfumes, fillers, and inorganic builders of the type such as carbonates, phosphates and silicates can also be present in the composition.

The soaps which are useful in the novel compositions of this invention are the so-called water soluble soaps of the soap-making art and include sodium, potassium, ammonium, and amine salts of the higher fatty acids; that is, those having about 8-20 carbon atoms per molecule. These soaps are normally prepared from such naturally-occurring esters as coconut oil, palm oil, olive oil, cottonseed oil, tung oil, corn oil, castor oil, soybean oil, wool fat, tallow, whale oil, and the like, as well as mixtures of these.

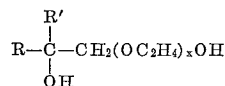
We claim:

1. An ethenoxy-substituted alkanol of the formula



wherein R and R' are selected from the group consisting of hydrogen and alkyl radicals, at least one of R and R' being an alkyl radical, said R and R' together having a total of from 6 to 24 carbon atoms, x is an integer of from 1 to 100, and Z is selected from the group consisting of chloro, hydroxyl and alkoxy radicals.

2. An ethenoxy-substituted alkanol of the formula



wherein R and R' are selected from the group consisting of hydrogen and alkyl radicals, at least one of R and R' being an alkyl radical, said R and R' together having a total of from 6 to 24 carbon atoms, and x is an integer of from 1 to 100.

References Cited by the Examiner

UNITED STATES PATENTS

2,197,467	4/1940	Evans et al.	260-615
2,302,121	11/1942	Harris	260-615
2,327,053	8/1943	Marple et al.	260-611
2,359,750	10/1944	Collins	260-615
2,379,703	7/1945	Geltner	260-615
2,380,185	7/1945	Marple et al.	260-615
2,543,744	3/1951	Fox	252-132
2,688,604	9/1954	Suen	260-615
2,875,153	2/1959	Dalton	252-132
2,934,568	4/1960	Barker	260-615

OTHER REFERENCES

McClelland et al.: Chemical & Engineering News, vol. 23 (1945), pp. 247-251.

LEON ZITVER, *Primary Examiner.*

JULIUS GREENWALD, CHARLES B. PARKER,
Examiners.