MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1964A
CONVENTION APPLICATION FOR STANDARD PATENT OR A STANDARD PATENT OF ADDITION

70511/81

Full name(s) of Applicant(s)
HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN
of Henkelstrasse 67, Dusseldorf, Germany
hereby apply for the grant of a standard patent
for an invention entitled
"AMPHOTENSIDES WITH INCREASED STORAGE STABILITY"

which is described in the accompanying complete specification.

DETAILS OF BASIC APPLICATION(s)
Number(s) of Basic Application(s)
P 30 18 201.5
Name(s) of Convention Country(ies) in which Basic Application(s) was/were filed
Federal Republic of Germany
Date(s) of Basic Application(s)
13th May, 1980 (respectively)

My/Our address for service is:
C/- SPRUSON & FERGUSON
PATENT ATTORNEYS
CBA CENTRE, 60 MARGARET ST.
SYDNEY, NEW SOUTH WALES.
AUSTRALIA.

Dated this EIGHTH day of MAY 1981

HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN

By: ____________________________
Registered Patent Attorney
DECLARATION IN SUPPORT OF A CONVENTION APPLICATION FOR A PATENT OR PATENT OF ADDITION

In support of the Convention Application made for a patent for an invention entitled

"AMPHOTENSIDES WITH INCREASED STORAGE STABILITY"

70511/81

1. Dr. Günter Schenck
   of Am Bühl 49
   4330 Mülheim / Germany

do solemnly and sincerely declare as follows:—

1. (or, in the case of an application by a body corporate)

   1. I am authorised by HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN
      the applicant for the patent

2. The basic application as defined by Section 141 of the Act was made in

   Federal Republic of Germany

   on the 13th day of May 1980 by

   HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN
   of Düsseldorf, Germany

3. Dr. Uwe Ploog of Haydnweg 6, 5657 Haan,
   Germany;
   Günter Uphues of Robert-Koch-Straße 45,
   4019 Monheim / Germany;
   and Manfred Petzold of Am Falder 93,
   4000 Düsseldorf 13 / Germany;

   are the actual inventors of the invention and the facts upon which the applicant is entitled to make the application are as follows:

   The said applicant is the assignee of the actual inventors.

4. The basic application referred to in paragraph 2 of this Declaration was the first application made in a Convention country in respect of the invention the subject of the application.

Declared at Düsseldorf this 23rd day of April 1981

[Signature of Declarant]

To:
The Commissioner of Patents,

Dr. Günter Schenck
SPRUSON & FERGUSON, SYDNEY.
Claim

1. A process for refining the crude condensation product of aminoalkylalkanolamines of the formula

\[ \text{H}_2\text{N}-\!(\text{CH}_2)_m-\text{NH}-\!(\text{CH}_2)_n-\text{OH} \]

wherein \( m \) is an integer of from 2 to 6 and \( n \) is 2 or 3, and fatty acids having from 6 to 22 carbon atoms, which comprises the step of subjecting the crude condensation product to alkaline hydrolysis.

11. A process for preparing amphotensides having increased storage stability which comprises the steps of:

(a) condensing an aminoalkylalkanolamine of the formula

\[ \text{H}_2\text{N}-\!(\text{CH}_2)_m-\text{NH}-\!(\text{CH}_2)_n-\text{OH} \]

wherein \( m \) is an integer of from 2 to 6 and \( n \) is 2 or 3, with fatty acids having from 6 to 22 carbon atoms;

(b) subjecting the condensation product of step (a) to alkaline hydrolysis; and

(c) subjecting the product from step (b) to alkylation and, optionally, quaternization.

Reactions with Sodium Chloroacetate

Example 4 - Without alkaline pretreatment
The following statement is a full description of this invention, including the best method of performing it known to me/us:

A clear solution with a solids content of approximately...
ABSTRACT OF THE DISCLOSURE

This invention relates to a process for refining the crude condensation product of aminoalkyalkanolamines of the formula

\[ \text{H}_2\text{N}-(\text{CH}_2)_m\text{-NH-(CH}_2)_n\text{-OH} \]

wherein \( m \) is an integer of from 2 to 6 and \( n \) is 2 or 3, and fatty acids having from 6 to 22 carbon atoms, which comprises the step of subjecting the crude condensation product to alkaline hydrolysis.

Example 8 - With alkaline pretreatment

The procedure of Example 7 was repeated with the exception
AMPHOTENSIDES WITH INCREASED STORAGE STABILITY

FIELD OF THE INVENTION

This invention relates to the refining of the crude condensation products of aminoalkylalkanol amines. More particularly, this invention relates to the refining of such condensation products and the preparation of amphotensides with increased storage stability.

BACKGROUND OF THE INVENTION

An important process for the preparation of amphotensides starts with condensation products of N-monosubstituted alkylenediamines that are alkylated in an additional reaction with alkylating agents, for example, sodium chloroacetate. Also, products based upon N-aminoethylethanolamines and fatty acids are important as tensides that protect the skin.

The preparation of such compounds is known principally from U.S. Patents Nos. 2,528,378, 2,528,379, and 2,528,380, incorporated herein by reference. While it had previously been assumed that alkylation or quaternization products of imidazoline were formed in this procedure, proof is available now that the products are mainly alkylation or quaternization products of aminoamides that are formed by hydrolysis.

Reactions with 2-Acrylamido-2-methylpropanesulfonic acid (AMPS)

Example 10 - Without alkaline pretreatment
of the imidazoline intermediate in the aqueous medium. Reference should also be made to, for example, German Published Application (DE-AS) No. 1,084,414, incorporated herein by reference.

The preparation of such tensides primarily involves the condensation of approximately 1 mol of fatty acid or fatty acid mixture with one mol of an aminoalkylalkanolamine—particularly aminoethylethanolamine—with a gradual increase in the temperature and, finally, under vacuum. This condensation product is then converted into the amphotenside with variable amounts of an alkylating agent, for example, sodium chloroacetate, usually in an aqueous alkaline solution. Details for these steps of the process are found in the patents mentioned above as well as in Dr. K. Lindner, "Tenside-Textilhilfsmittel-Waschrohstoffe", Wissenschaftliche Verlags GmbH, Stuttgart, 1964, pages 1041 and 1042, German Published Application (DE-OS) No. 27 52 116, published European Patent Application No. 001,006, and U.S. Patents Nos. 2,773,068, and 3,408,361 incorporated herein by reference.

The quality of the amphotensides obtained, and particularly their storage stability, are dependent upon the purity of the imidazoline derivative obtained in the first condensation step. The reaction between fatty acids and amino-

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Example 13 - With alkaline pretreatment

The procedure of Example 10 was followed, except that the suspension was agitated for one hour at 80° to 90° C before the addition of chlorohydropyrone-sulfonic acid.
alkylalkanolamines proceeds toward more than merely the formation of the desired imidazoline derivative, and multiple secondary reactions complicate the reaction process. The significance of this aspect has been emphasized again recently. For example, in E. G. Lomax, "New and Improved Balanced Amphotericcs", Manufacturing Chemist and Aerosol News, Vol. 50, No. 8, August 1979, pages 39 and 41, it is disclosed that the undesirable secondary reactions in the step of the imidazoline formation can be suppressed with an excess of aminoethylethanolamine but that this excess must be removed by distillation at the end of the reaction. However, aminoethylethanolamine itself can introduce new difficulties by cyclization to the piperazine.

The purity of the imidazoline obtained in the first reaction step has a decisive influence on the storage stability of the amphotensides obtained by subsequent alkylation of their aqueous solutions. Even minor impurities lead to the separation of a solid phase, in the form of turbidity or sediments, after a shorter or longer storage period. Such products are not suitable for practical application or are at the least of limited value.

OBJECTS OF THE INVENTION

It is an object of the invention to provide an improved process for refining the crude condensation products of amino-alkylalkanolamines.
It is also an object of the invention to provide ampho-
tensides that have increased storage stability.

It is a further object of the invention to provide a
process for the refining of the crude condensation products
of aminoalkylalkanolamines of the formula

\[ \text{H}_2\text{N} - (\text{CH}_2)_m - \text{NH} - (\text{CH}_2)_n - \text{OH} \]

wherein \( m \) is an integer of from 2 to 6 and \( n \) is 2 or 3, with
fatty acids having from about 6 to 22 carbon atoms, wherein
the crude condensation product is subjected to alkaline hy-
drolysis.

These and other objects of the invention will become
more apparent from the description below.

DETAILED DESCRIPTION OF THE INVENTION

Applicants have surprisingly developed a procedure for
obtaining a condensation product that is as pure as possible,
that does not employ the circuitous routes now used.
According to the invention, it is possible to eliminate in-
terfering by-products in the simplest possible manner by
introducing a simple refining step for the primarily obtained
condensate.

Applicants have found that the crude condensation product
of fatty acids and aminoalkylalkanolamines can be converted

The claims defining the invention are as follows:

1. A process for refining the crude condensation pro-
into a purified product by a simple alkaline hydrolysis and that this procedure yields improved amphotensides with particularly increased storage stability upon subsequent alkylation and, optionally, quaternization. Accordingly, the invention relates to a process for the refining of the crude condensation product of aminoalkylalkanolamines of the formula

\[ \text{H}_2\text{N}-\left(\text{CH}_2\right)_{m}-\text{NH}-\left(\text{CH}_2\right)_{n}-\text{OH} \]  

wherein \( m \) is an integer of from 2 to 6, particularly 2, 3, or 6, and \( n \) is 2 or 3, particularly 2, with fatty acids having from about 6 to 22 carbon atoms, wherein the crude condensation products of aminoalkylalkanolamines and fatty acids are subjected to alkaline hydrolysis. The invention also relates to the subsequent alkylation and, optionally, quaternization of the refined condensation product into amphotensides with increased storage stability.

In the process according to the invention, the amount of alkali used in the alkaline hydrolysis preferably is adjusted to the amount of diamide of the formula

\[ \text{R}-\text{CO-NH-}\left(\text{CH}_2\right)_{m}-\text{N-CO-R} \]

\[ \left(\text{CH}_2\right)_{n}-\text{OH} \]  

wherein \( m \) and \( n \) are as defined above, present in the crude condensate. The amount of alkali in this process step is especially preferably chosen so that it is present in a quantity at least approximately equimolar to the diamide in

8. The process of Claim 7, wherein the alkaline hydrolysis is carried out at temperatures of from about 80° to 90° C.
the crude condensation product.

An underlying basis of the invention is that the content of interfering by-products in the finished tensides can be significantly reduced when the condensation product of fatty acid and amine is subjected to an alkaline pretreatment in an aqueous medium prior to a further reaction. Experiments have particularly demonstrated that the diamide formed during the reaction is cleaved quantitatively at the tertiary amide group by aqueous alkali solution to form the monoamide of the primary amino group and fatty acid. The fatty acid forms soap in the refining treatment according to the invention, which soap may remain in the reaction mixture.

It is actually very surprising that the difficulties with respect to the storage stability of the amphotensides ultimately obtained, which so far could be corrected only with relatively costly refining steps, are eliminated by the intermediary treatment according to the invention. Considering the fact that the alkylation and, optionally, quaternization of the reaction product obtained in the first step are carried out in the aqueous alkaline medium — for example, with sodium chloroacetate — and that the temperature and alkalinity conditions of the process are chosen so that they are at least comparable to the conditions of the
intermediate step according to the invention, this result is the more surprising. It could not be expected that the separate alkaline hydrolysis according to the invention of the crude condensation product from the first process step could yield better results than a corresponding aqueous-alkaline treatment during the course of the alkylation or quaternization of the condensation product.

However, the alkaline pretreatment according to the invention results in products that remain clear, even after dilution, for more than six months. Costly refining steps such as recovery of the excess amine or distillation of the imidazoline are no longer necessary.

The alkaline hydrolytic treatment according to the invention preferably takes place in the temperature range of from about 70 °C to 100 °C, especially in the temperature range of from about 80 °C to 90 °C. Particularly suitable as alkalies are alkali metal hydroxides, especially sodium hydroxide. The amount of alkali is preferably in the range of from about 1 to 3 times the equimolar alkali requirements, based on the diamide present in the condensation product. Preferably alkali amounts in the range of from about 1 to 2 equivalents, based on the diamide, are used. The crude condensation product is advantageously suspended in an amount of water corresponding to from about 0.5 to 10 times, especially from about 1 to 5 times, the quantity of
crude condensation product for the alkaline hydrolysis according to the invention. The aqueous alkaline hydrolysis is allowed to continue to the practically complete removal of the diamide, which is present as by-product. The diamide content of the crude product and its decrease during the treatment according to the invention can be determined by a known method, for example, by working with ion exchangers.

The aminoalkylalkanolamines used according to the invention are those of Formula II. The compound aminoethyl-ethanolamine is the starting amine of Formula II that has the greatest practical significance. The fatty acids used for the condensation have from about 6 to 22 carbon atoms, preferably from about 8 to 18 carbon atoms. The fatty acids may be present as pure components or as mixtures of fatty acids having different chain lengths. They may be natural and/or synthetic in origin.

The condensation product pretreated by alkaline hydrolysis according to the invention is then converted to the finished amphotenside by a known method. Such methods are described in the references cited above.
The pretreatment according to the invention is valuable not only for the preparation of the so-called imidazolinium tensides but for other tensides as well. Clear products with a long storage life are also obtained by the conversion of the alkali-treated condensation products of amines with alkylating agents of such as chlorohydroxypropanesulfonic acid or propanesulfone, as well as by alkylation with vinyl group-containing compounds such as acrylic acid or acrylic acid ester, with subsequent saponification and 2-acrylamido-2-methylpropanesulfonic acid or the corresponding alkali metal salts.

The amphotensides may contain alkylated and optionally quaternary nitrogen formed in the second reaction step, depending on the structure and the degree of conversion. Another embodiment of the invention includes the preparation of such amphotensides with the use of a product that has been treated by the described alkaline hydrolytic process according to the invention.

The following examples are intended to illustrate the invention and should not be construed as limiting the invention thereto. The percentages given in the examples are in percent by weight, based on the weight of the total condensation product. The diamide content was determined in the following manner:
The condensation product in the form of an alcoholic solution was led over a highly acidic ion exchange resin. The eluate was evaporated, and the ratio of the residue to the weighed-in-amount of condensation product introduced was determined. The amount of diamide was calculated from the determination of the acid number and the total nitrogen.

EXAMPLES

Condensation Products of Fatty Acids with Aminoethylethanolamine

Example 1 - Molar ratio of 1:1

In a three-neck flask with agitator, nitrogen inlet, thermometer, and distillation attachment, 208 gm (1 mol) of C_{8/18} coconut oil acid and 104 gm (1 mol) of aminoethylethanolamine were mixed and slowly heated to 200 °C, over a period of approximately six hours. After a total of 20 gm of water containing amine was distilled off, approximately 290 gm of a slowly congealing, yellow mass were obtained, which had the following analytical data:

- Acid No : 2.8
- N_{Kj} : 9.5%
- N_{titr.} : 4.7%
- Diamide content : 16% (= 0.04 mol/100 gm).

A UV-spectroscopy analysis at 230 mm indicated an imidazoline content of 8.0%.
Example 2 - Molar ratio 1:1.1

Following the procedure described for Example 1, 208 gm (1 mol) of coconut oil acid were condensed with 114.5 gm (1.1 mol) of aminoethylethanolamine. Approximately 300 gm of a slowly congealing, yellow mass were obtained, which had the following analytical data:

<table>
<thead>
<tr>
<th>Component</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid No.</td>
<td>2.3</td>
</tr>
<tr>
<td>$N_{Kj}$</td>
<td>10.2%</td>
</tr>
<tr>
<td>$N_{titr}$</td>
<td>5.6%</td>
</tr>
<tr>
<td>Diamide content</td>
<td>10.7% ($\approx$ 0.02 mol/100 gm)</td>
</tr>
<tr>
<td>Imidazoline content</td>
<td>3.0%</td>
</tr>
</tbody>
</table>

Example 3 - Molar ratio of 1:1.5

In accordance with the procedure described for Example 1, 208 gm (1 mol) of coconut oil acid were condensed with 156 gm (1.5 mol) of aminoethylethanolamine with slow heating, so that the content of free amine remained as high as possible during the reaction. The reaction temperature at the end was up to 180°C, at approximately 14 mbar. After the excess amine was distilled off, approximately 270 gm of residue were left, which had the following analytical data:

<table>
<thead>
<tr>
<th>Component</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$N_{Kj}$</td>
<td>10.3%</td>
</tr>
<tr>
<td>$N_{titr}$</td>
<td>5.2%</td>
</tr>
<tr>
<td>Diamide content</td>
<td>2.7% ($\approx$ 0.006 mol/100 gm)</td>
</tr>
<tr>
<td>Imidazoline content</td>
<td>96%</td>
</tr>
</tbody>
</table>
Reactions with Sodium Chloroacetate

Example 4 - Without alkaline pretreatment

A dispersion was prepared with 90 gm (0.3 mol, calculated from N titr.) of product from Example 1 in 148 gm of water and reacted with 203 gm (0.7 mol) of freshly prepared 40% solution of sodium chloroacetate, at 60 °C. The pH rose to 11.52 upon the addition of 56 gm (0.7 mol) of 50% sodium hydroxide solution. The mixture was agitated for two hours at that temperature, which was then increased to 80 °C for one hour. The pH slowly dropped to about 10. The approximately 40% solids product began to become turbid after two days at room temperature.

Example 5 - Without alkaline pretreatment

A dispersion was prepared with 30 gm (0.32 mol, calculated from N titr.) of product from Example 2 in 134 gm of water and reacted with 214 gm (0.74 mol) of a freshly prepared 40% sodium chloroacetate solution, at 60 °C. Then, 58.8 gm (0.74 mol) of 50% sodium hydroxide solution were added at 60 °C. The mixture was kept at 60 °C for two hours, and then the temperature was increased to 80 °C. The pH rose to 11.5 after the addition of the sodium hydroxide solution and dropped slowly to 10.1 after one hour of heating at 80 °C.
The following statement is a full description of this invention, including the best method of performing it known to us.

A clear solution with a solids content of approximately 40% was obtained, which solution began to become turbid after only two days at room temperature, however.

**Example 6 - Without alkaline pretreatment**

A dispersion was prepared with 90 gm (0.3 mol)! of imidazoline from Example 3 in 134 gm of water. This was kept at 60 °C for one hour and then reacted with 203 gm (0.7 mol) of a freshly prepared 40% sodium chloroacetate solution. Next, 56 gm (0.7 mol) of 50% sodium hydroxide solution were added, whereupon the pH rose to 11.6. The mixture was agitated at this temperature for two hours and for an additional hour at 80 °C. After approximately 10 weeks of storage at room temperature, the first signs of turbidity appeared.

**Example 7 - With alkaline pretreatment**

A dispersion was prepared with 90 gm (0.3 mol, calculated from N_titr.) of product from Example 1, which contained 0.035 mol of diamide, in 148 gm of water, reacted with 1.2 gm (0.015 mol) of 50% sodium hydroxide solution, and agitated for one hour at 80 °C to 90 °C. Then the process was continued as described in Example 4, the product obtained being as clear as that obtained in Example 4. However, the product also became turbid after six days because not all the diamide was saponified.
Example 8 - With alkaline pretreatment

The procedure of Example 7 was repeated with the exception that 4.8 gm (0.06 mol) of 50% sodium hydroxide solution were reacted with the product from Example 1. The diamide was quantitatively saponified to the monoacyl product, as was also apparent from the analysis. The product remained clear for more than six months.

Example 9 - With alkaline pretreatment

A dispersion was prepared with 80 gm (0.32 mol) of product from Example 2 in 60 gm of water, reacted with 4.8 gm (0.06 mol) of 50% sodium hydroxide solution, and agitated for one hour at 80°C. After further conversion according to the procedure described in Example 5, a tenside was obtained that remained clear for more than six months.

It is apparent from Examples 4 to 6 as well as Examples 7 to 9 that even the use of a very pure imidazoline in the first step (see Example 6) does not result in the improvement of the quality, such as is obtained with starting materials rich in diamide (Examples 1 and 2), after alkaline pretreatment. Advantageously sodium hydroxide is used in an amount that is at least equivalent, but preferably approximately double, the quantity necessary on the basis of the diamide content.
Reactions with 2-Acrylamido-2-methylpropanesulfonic acid (AMPS)

Example 10 - Without alkaline pretreatment

A suspension was prepared with 75.0 gm (0.3 mol, calculated from N_titr.) of product from Example 2 in 200 gm of water and reacted with 80.6 g (0.39 mol) AMPS and then with 31.2 gm (0.39 mol) of 50% sodium hydroxide solution. The mixture was agitated for four hours at 80 °C. The finished product was a clear, thin liquid; however, precipitation was noticed after two days.

Example 11 - With alkaline pretreatment

A suspension was prepared with 75.0 gm (0.3 mol) of product from Example 2 in 200 gm of water and agitated for one hour with 4.8 gm (0.06 mol) of 50% sodium hydroxide solution at 80 °C to 90 °C. Further conversion proceeded as in Example 10. The product remained completely clear even after more than six months.

Reactions with Chlorohydroxypropanesulfonic acid (Sodium salt)

Example 12 - Without alkaline pretreatment

A dispersion prepared from 75 gm (0.3 mol) of product from Example 2 in 200 ml of water was reacted with 63.6 gm (0.3 mol) of chlorohydroxypropanesulfonic acid (sodium salt), for 30 minutes at 80 °C, and with 24.0 gm (0.3 mol) of 50% sodium hydroxide solution. The mixture was then agitated for four hours at 50 °C. The end product was clear, but became turbid after three days.
Example 13 - With alkaline pretreatment

The procedure of Example 10 was followed, except that the suspension was agitated for one hour at 80° to 90°C before the addition of chlorohydroxypropanesulfonic acid (sodium salt), after the addition of 4.8 gm (0.06 mol) of 50% sodium hydroxide solution. The product remained clear after more than six months.

Reactions with Acrylic Acid

Example 14 - Without alkaline pretreatment

Seventy-five grams (0.3 mol) of product from Example 1 and 21.6 gm (0.3 mol) of acrylic acid were mixed and agitated for one hour at 80°C and then diluted with 145 gm of water. The product began to become turbid after two weeks.

Example 15 - With alkaline pretreatment

Seventy-five grams (0.3 mol) of product from Example 1 and 4.8 gm (0.06 mol) of 50% sodium hydroxide solution were agitated for one hour at 80°C and the treated further according to the procedure of Example 14. The product remained clear for more than six months.

The preceding specific embodiments are illustrative of the practice of the invention. It is to be understood, however, that other expedients known to those skilled in the art or disclosed herein, may be employed without departing from the spirit of the invention or the scope of the appended claims.
The claims defining the invention are as follows:

1. A process for refining the crude condensation product of aminoalkylalkanolamines of the formula

$$H_2N-(\text{CH}_2)_m-\text{NH}-(\text{CH}_2)_n-\text{OH}$$

wherein \( m \) is an integer of from 2 to 6 and \( n \) is 2 or 3, and fatty acids having from 6 to 22 carbon atoms, which comprises the step of subjecting the crude condensation product to alkaline hydrolysis.

2. The process of Claim 1, wherein \( m \) is 2, 3, or 6.

3. The process of Claim 1, wherein \( n \) is 2.

4. The process of Claim 1, wherein the amount of alkali used in the alkaline hydrolysis is in proportion to the amount of diamide present in the crude condensation product.

5. The process of Claim 4, wherein the alkali is used in an equimolar amount.

6. The process of Claim 4, wherein the amount of alkali does not exceed 3 times the molar amount of diamide.

7. The process of Claim 1, wherein the alkaline hydrolysis is carried out at temperatures of from about 70° to 100° C.
8. The process of Claim 7, wherein the alkaline hydrolysis is carried out at temperatures of from about 80° to 90°C.

9. The process of Claim 1, wherein fatty acids or mixtures of fatty acids having from 6 to 20 carbon atoms are used.

10. The process of Claim 9, wherein fatty acids or mixtures of fatty acids having from 8 to 18 carbon atoms are used.

11. A process for preparing amphotensides having increased storage stability which comprises the steps of:

(a) condensing an aminoalkylalkanolamine of the formula

\[ \text{H}_2\text{N}-(\text{CH}_2)_m-\text{NH}-(\text{CH}_2)_n-\text{OH} \]

wherein m is an integer of from 2 to 6 and n is 2 or 3, with fatty acids having from 6 to 22 carbon atoms;

(b) subjecting the condensation product of step (a) to alkaline hydrolysis; and

(c) subjecting the product from step (b) to alkylation and, optionally, quaternization.

DATED this SEVENTH day of MAY, 1981
HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN

Patent Attorneys for the Applicant
SPRUSON & FERGUSON