

[54] N-OXIDE-IMINODICARBOXYLATES

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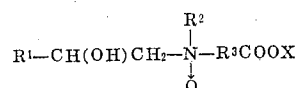
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[57]

ABSTRACT

Novel oxides of aminocarboxylates or aminocarboxylic acids are of the formula:



wherein R^1 is an aliphatic hydrocarbon radical of four to 20 carbon atoms, R^2 is a hydrocarbon radical of one to four carbon atoms or is R^3COOX , R^3 is a divalent aliphatic or aromatic hydrocarbon radical of one to nine carbon atoms, which may be the same as or different from another R^3 radical which may be present in the compound, X is hydrogen, alkali metal, alkaline earth metal, other suitable salt-forming metal, ammonium, alkylamine or alkanolamine, which may be the same as or different from any other X in the formula, with it being understood that in the case of divalent or trivalent X's it may be joined to a plurality of the shown organic moieties through the carboxylic oxygen(s) thereof.

The novel compounds are possessed of surface activity and are substantive to fibrous materials, e.g., cotton and wool. They may be used as detergents or components of detergent compositions and serve as softeners for fibrous materials.

5 Claims, No Drawings

N-OXIDE-IMINODICARBOXYLATES

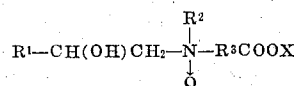
This invention relates to novel N-oxides of aminocarboxylates or aminocarboxylic acids in which an amino hydrogen is substituted by a 2-hydroxyalkyl group and either an alkyl group or a hydrocarbyl carboxylate or acid. Such compounds have been found to be useful surface active agents and are substantive to various fibrous materials. Thus, they serve as: emulsifiers for oils, to form oil-in-water emulsions; solubilizing agents for lipophilic materials; wetting agents; detergents; and softeners for textiles.

Since the advent of commercial synthetic detergents and emulsifiers to replace the water-soluble higher fatty acid soaps, continued research work has been performed with the object of discovering detergents and other compounds that are useful to improve laundering of textiles. A wide variety of types of surface active agents and detergents has been produced in the laboratory and many of these have been commercially manufactured. Although initially, primary interest was directed to improving detergency and hard water cleaning characteristics, with the substantial attainment of such objectives increased research has been expended to make aesthetically satisfactory improved detergent compositions which are relatively inexpensive and which perform other functions in washing operations, in addition to cleaning. Thus, detergent products containing bleaches, brighteners, soil-suspending chemicals, protective colloids, preservatives, antioxidants, anti-tarnish agents, foaming agents, improved perfumes, builders and solubilizers have been produced and have met with good consumer acceptances. In many cases, the compositions made included additional ingredients which added their functions to those of the detergent constituent. At other times, builders or surface active materials improved the detergency of the principal detergent constituent. Also, synergistic combinations of detergents were discovered. Although such combination products are useful and many have been successfully marketed, efforts have been made to improve laundering operations further by compounds which in themselves possess favorable qualities, in addition to their deterative abilities. Thus, attempts have been made to create detergents which clean and at the same time sequester the hardness ions of various types of hard waters. Even when such products were successful, it was often found that further work had to be done to make them into detergents useful to wash well in both hard and soft waters. Attempts have been made to produce detergents which themselves also impart brightening effects to clothes washed, or have bleaching properties. Another example of a dual function detergent is one which is germicidal so that the laundered material is made substantially germ-free in the washing process. Despite the fact that in some cases successful dual action detergents have been produced, most commercial detergent formulas today do not include substantial proportions of such compounds and to obtain special properties, specific additives are usually employed.

The present invention is of compounds which have effective detergent action, making them useful in removing dirt or various stains from clothing or other articles to be laundered, while at the same time, in a preferred aspect of the invention, they are sufficiently

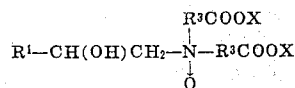
substantive to various textiles to cause deposition on the textiles and retention thereon of the detergent or a portion of it, which serves to "soften" the textile, giving it a feel that is pleasant to the touch. Of course, softeners have been used in treating textiles but usually they are deposited on the textile by immersing it in a solution of the softening compound after completion of the washing operation. In cases where such compounds are applied earlier, the softeners must possess sufficient substantivity to allow a part thereof to remain on the textile and have its effects discernible to the user after washing. It is generally observed that detergents oppose the deposition of substantive material on textiles and usually only small fraction of the substantive material present remains on the material washed, due to the action of the detergent. For this reason, the production of a deterative and textile-softening compound of useful activities had appeared unlikely. However, the present compounds possess significant deterative properties and in preferred embodiments, are substantive to textiles and impart a soft "hand" thereto. Additionally, they are generally low foaming materials and may possess anti-static properties, both of which characteristics are often highly desirable in detergent-softeners for textiles.

In accordance with the present invention there are provided novel compounds of the formula:



which are effective detergents and which preferably also serve to soften fibrous materials when applied to them, as in aqueous solutions of the compounds. In the formula, R^1 is an aliphatic hydrocarbon radical of four to 20 carbon atoms, R^2 is a hydrocarbon radical of one to four carbon atoms or is R^3COOX , and R^3 is a divalent aliphatic or aromatic hydrocarbon radical of one to nine carbon atoms. X is hydrogen, alkali metal, alkaline earth metal or suitable salt-forming metal or may be ammonium, alkylamine or alkanolamine, either mono-, di- or tri-alkylamine or -alkanolamine. The alkyl and alkanol groups of the salt-forming amines are of one to four carbon atoms, preferably two to three carbon atoms. Both R^3 and X may be the same or different from any other R^3 or X in the compounds, or X in the compounds. It should be understood that X signifies a monovalent element or radical or a bond or valence of a divalent or trivalent element or radical. In preferred compounds X is hydrogen, alkali metal or other monovalent ion.

When R^2 is R^3COOX the product is an N-oxide-iminodicarboxylate of the formula



Preferred embodiments of the invention are those amine oxides of the formula previously given wherein R^1 is an aliphatic hydrocarbon radical of eight to 14 carbon atoms, R^2 is an aliphatic hydrocarbon radical of one to two carbon atoms or is R^3COOX , R^3 is a divalent aliphatic hydrocarbon radical of one to three carbon atoms, X is hydrogen or a monovalent salt-forming ion which is either an alkali metal, ammonium, mono-, di-

or tri-alkylamine, or mono-, di-, or tri-alkanolamine. In such compositions, it is preferred for both X's and R³'s to be the same, if more than one should be present.

The preferred compounds mentioned above are found to be excellent textile softeners, especially for cotton articles washed with them, even in the presence of builders and after normal rinsing. With respect to such properties, the most preferred compounds are the salts of N-(2-hydroxy-higher alkyl)-N-methyl glycine-N-oxides and the N-(2-hydroxy-higher alkyl)-aminodiacetic acid-N-oxides, -N-oxides, wherein the higher alkyl is of 12 to 16 carbon atoms and is of straight chain structure. Of such compounds, it is preferred to employ the sodium and potassium salts, with the sodium salt being that which is most useful. Of the iminodiacetic acid compounds and the N-methyl glycine compounds mentioned, the more effective for the softening of cotton textiles are the N-methyl glycines.

Although preferred and most preferred embodiments of the invention have been mentioned above, which compounds have exceptionally good softening activities for textiles, other compounds within the scope of the present invention are useful surface active agents, wetting agents, emulsifiers and detergents, although they might not also produce the same extent of the extremely desirable softening activity shown by the most preferred materials, and might even be essentially ineffective in this respect, in some cases.

Among the compounds within the scope of the generic formula previously given are N-(2-hydroxy-n-butyl)-N-butyl glycine-N-oxide, sodium salt; N-(2-hydroxy-n-octyl)-N-isopropyl glycine-N-oxide, potassium salt; N-(2-hydroxydodecyl)-N-t-butyl glycine-N-oxide; N-(2-hydroxytetradecyl)-N-ethyl glycine-N-oxide, ammonium salt; N-(2-hydroxypropylene tetramer)-N-methyl glycine-N-oxide, triethylamine salt; N-(2-hydroxyhexadecyl)-N-ethyl glycine-N-oxide, triethanolamine salt; N-(2-hydroxyoctadecyl)-N-methyl glycine-N-oxide, diethanolamine salt; N-(2-hydroxyoctadecenyl)-N-n-propyl glycine-N-oxide, di-n-butylamine salt; N-(2-hydroxy-2,2-dimethyl-ethyl)-iminodiacetic acid-N-oxide, mono-n-propanolamine salt; N-(2-hydroxyhexadecyl)-iminodiacetic acid-N-oxide, disodium salt; N-(2-hydroxy-n-hexyl)-iminodipropionic acid-N-oxide, monopotassium salt; N-(2-hydroxy-6-ethyl-n-decyl)-iminodipropionic acid-N-oxide, di-triethanolamine salt; N-(2-hydroxydodecyl)-iminodipropionic acid-N-oxide, magnesium salt; N-(2-hydroxyhexadecyl)-iminodiacetic acid-N-oxide, magnesium salt; N-(2-hydroxyoctadecenyl)-iminobenzoic-toluic acid-N-oxide, diisopropylamine salt; N-(2-hydroxyoctadecyl)-iminodipentanoic acid-N-oxide, mixed sodium and potassium salt; N-(2-hydroxytetradecyl)-N-methyl glycine-N-oxide; N-(2-hydroxydodecyl)-N-ethyl glycine-N-oxide; and N-(2-hydroxyhexadecyl)-iminodiacetic acid-N-oxide. The alkyl groups of the N-(2-hydroxyalkyl) portions of the above compounds, unless otherwise indicated, are the preferred straight chain groups, terminally joined to the nitrogen atom.

The above compounds are only illustrative of those of the formulas previously given but other compounds within the scopes of the formulas are also useful and in many cases possess properties superior to compounds

specifically listed above. Thus, variations in the above specific formulas may be made wherein other mentioned groups are substituted for the groups shown. In some cases, additional non-interfering substituents may be employed, usually on the longer chain portions of the molecules, where they do not significantly affect the proportions of the final product. Among such substituents may be mentioned amino, hydroxy, halogen, e.g., chlorine, bromine, fluorine, and hydroxy-lower alkyl, wherein the alkyl is of up to four carbon atoms. Such substituents will usually be present in small number, generally no more than four being present on the molecule and usually less than three. Generally it is preferable that the compounds not be substituted but the substituted compounds are often of utility similar to those which are literally within the formulas given. As illustrated in the recitations of specific compounds within the formulas, given above, mixed salts may be employed, as may be acid salts. The salt-forming metal or other radical, if divalent or polyvalent, may be joined to both acid portions of the imino compounds. Also, in the formation of the salts, a single salt-forming ion may be joined to two molecules of the acids of this invention.

Although the normal alkyl groups are preferred for R¹, branched chain materials may also be used. Thus, the various alkyl groups formed from propylene may be employed, such as the propylene tetramer and pentamer, a preferred form of which is a mixture averaging 13 carbon atoms. Also, although saturated hydrocarbons are preferred, those which are unsaturated, to the extent of one or two double bonds per radical, may also function as R¹. R² should be a short chain material, preferably unsubstituted, and is most preferably methyl. When such lower alkyl group is replaced by hydrogen, the product obtained does not have the desirable detergent and softening properties of the compounds of this invention and is difficult to manufacture. R³ is preferably a short chain alkylene, usually of one to two carbon atoms, but it has been found that longer chain alkenes and even divalent hydrocarbyl aromatic compounds make useful products. X, while it may be hydrogen, is preferably a salt-forming ion. This is so because the salt is usually more stable and free-flowing, and the product is most frequently employed in alkaline solutions. Of the salt-forming ions those which are monovalent are preferred, because of their generally greater water solubility, an important feature in the use of the present compounds. However, even though water solubility may be low, other salts can be employed as washing agents in other polar media, and even may be useful in an aqueous medium.

The novel compounds of this invention may be prepared by oxidation of the corresponding amine. Thus, the N-(2-hydroxyalkyl)-aminocarboxylic acid or iminodicarboxylic acid or salt thereof may be treated with an oxidizing agent, such as hydrogen peroxide or ozone, preferably in aqueous solution, and the water remaining after the reaction may be removed by any suitable technique, e.g., freeze drying. Usually, the reaction will be run in an aqueous solution of a soluble salt of the starting material and will be conducted by slowly admixing the oxidizing agent with the solution of salt at a temperature about room temperature, with cooling to maintain such temperature or one slightly

higher sometimes being desirable because of the exothermic reaction and generation of excess heat. Cooling will be most useful when there is present a high concentration of amine to be oxidized. Normally, the addition of the oxidizing agent will take place within a period of from 30 seconds to 1 hour, at a temperature of 15° to 50°C. The solution may be held for an additional 30 minutes to 18 hours at a temperature of 40° to 90°C., to decompose excess H_2O_2 , but this can also be done with catalysts, such as platinum or palladium, or with reducing agents, such as sodium sulfite. The proportions of reactants employed are such that the oxidizing agent is initially present in excess. Such excess will usually be from 5 percent to 100 percent of the stoichiometric proportion, so as to aid in forcing the reaction and producing a 100 percent yield of the oxide. Of course, less than the stoichiometric proportion of oxidizing agent may be used, in which cases yields will suffer. It is normally not desirable to use less than 75 percent of the stoichiometric quantity of oxidizing agent, unless it is desired to make a mixture of the oxide or salt and the unoxidized aminocarboxylic acid or iminodicarboxylic acid or salt. Instead of utilizing the water soluble salt, a comparable reaction may be run with the acid, which may be solubilized by suitable solvents, e.g., ketones, such as acetone, methyl ethyl ketone. Also, the salts may be made by subsequent treatment of the acid oxides produced with alkaline material, e.g., NaOH, triethanolamine. The acid forms of the oxide may be produced by acidification of previously manufactured salts.

N-(2-hydroxyalkyl)-N-substituted aminocarboxylic acid or the corresponding iminodicarboxylic acid starting materials or salts may be made by reactions similar to those described in our co-pending applications entitled N-(2-Hydroxy-Higher Hydrocarbyl)-N-Lower Hydrocarbyl-Aminocarboxylates and N-(2-Hydroxyhydrocarbyl) Iminodicarboxylates, filed on the same day as this application. In brief, such reactions are of the N-substituted aminocarboxylic acid with hydrocarbon-1,2-epoxide. Such reactions are usually conducted at an elevated temperature, often about the boiling point of the reaction mixture, which may contain some water, and after the reaction has been completed, which may take from 10 minutes to 5 hours, the reaction mix is cooled and water is removed by any suitable technique. Such removal may be by freeze drying, flash vaporization, evaporation or other method. To remove impurities, the product may be extracted with a solvent for these, e.g., acetone. If desired, it may be recrystallized from alcohol. The starting epoxides and aminocarboxylic acids or salts are well-known compounds and methods for their manufacture are obvious.

In use, the present materials are normally employed in aqueous solution and are used either alone or with additives to make deterative solutions or emulsions and to act as wetting agents. They are also used to soften fabrics, especially cotton textiles. In some processes the invented compounds usefully wash and soften textiles in the same operation. The effectiveness of the invented materials, especially those most preferred, in softening textiles is very much unexpected, inasmuch as the present compounds help to release other materials which are adherent to the textile fibers. The very

softening effect obtained appears to depend upon substantivity of the invented compounds to the fiber. Also, such a substantivity appears to be effective with respect to vegetable and animal fibers, as well as various synthetic fibers. Therefore, the present compounds are highly advantageous for use in detergents and softening agents, and are especially suitable for use in combination detergent-softening compositions.

The following examples are given to illustrate the invention but are not to be considered as limitative. Unless otherwise stated, all parts given are by weight.

EXAMPLE 1

Fifteen parts of N-(2-hydroxydodecyl)-N-methyl glycine, sodium salt, are dissolved in 85 parts water and 7.0 parts of a 30% hydrogen peroxide solution in water are added slowly thereto. Such addition is dropwise, with both the glycine compound and the hydrogen peroxide being at 40°C. After completion of the addition of the hydrogen peroxide which takes about 5 minutes, the resulting aqueous solution is held at an average temperature of 75°C. for four hours, with the temperature variation being between 70°C. and 80°C. during this time. At the end of the four hour period, the solution is tested for the presence of peroxide and a negative result is obtained. Then, the solution is frozen and water is removed from it by a freeze drying operation, at an absolute pressure of 3 millimeters of mercury. The product remaining is a white, hygroscopic powder, identified as N-(2-hydroxydodecyl)-N-methyl glycine-N-oxide, sodium salt. The yield obtained is 15.5 parts, almost the stoichiometric amount.

Following the procedure the corresponding oxides are made from N-(2-hydroxytetradecyl)-N-methyl glycine, sodium salt; N-(2-hydroxyhexadecyl)-N-methyl glycine, sodium salt and N-(2-hydroxyoctadecyl)-N-methyl glycine, sodium salt. The only changes made in the procedure are in the use of the stoichiometrically adjusted proportions of the glycine compounds, with more of such compounds being used than in the above specific example, because of the greater molecular weights of the reactants. The products obtained are also white solids and are hygroscopic.

In addition to the sodium salts of the N-(2-hydroxyalkyl)-N-methyl glycine-N-oxides, corresponding oxides are made by utilizing ozone as a source of reactive oxygen. Again, addition is slow and the ozone may be diluted with inert gas so that it comprises from 5 to 50 percent by volume of such gas mixture. In place of ozone, other oxygen-releasing compounds may be used, such as the per-compounds, e.g., sodium perborate, peracetic acid, urea hydrogen peroxide, and other known and suitable oxidizing agents useful for the making of amine oxides. Such compounds are generally used in aqueous solution, with the proportion of available oxygen present corresponding to that described in the above example.

In addition to the sarcosine compounds, corresponding N-(2-hydroxy-alkyl)-N-substituted carboxylic acids and salts are used in replacement of the specific compound shown in the above example. Stoichiometrically equivalent proportions of N-(2-hydroxy-n-butyl)-N-n-propylamine-n-propionic acid, potassium salt; N-(2-hydroxy-n-decyl)-N-aminobenzoic acid, ammonium

salt; N-(2-hydroxyhexadecyl)-N-methyl-aminotoluic acid, triisopropylamine salt; and N-(2-hydroxydodecyl)-N-amyl-aminocetic acid, calcium salt are employed in replacement of the 15 parts of N-(2-hydroxydodecyl)-N-methyl glycine, sodium salt, and corresponding oxidations are effected by following the procedure of Example 1, to make the respective salts. They are useful surface active compounds, which may be employed as surface tension lowering agents, emulsifiers, detergents or textile softeners. When acidified, such compounds and those previously described in this example, are converted to the acid form. The corresponding acids also possess similar properties.

EXAMPLE 2

208.5 parts of N-(2-hydroxyhexadecyl)-iminodiacetic acid, disodium salt, are dissolved in 800 parts of water, at room temperature. The starting material is made by reaction of hexadecane-1,2-epoxide with the disodium salt of iminodiacetic acid. Subsequently, 700 parts of a 30 percent aqueous solution of hydrogen peroxide are slowly added to the iminodiacetate. The reaction mixture is heated at a temperature of 70° to 80°C. for 2½ hours. Then one part of palladium on charcoal is added and the reaction mixture is allowed to stand at room temperature for 18 hours, after which the insoluble materials are filtered off at room temperature and the product, a white powder, is obtained by freeze drying the filtered solution. About 210 parts of product are obtained.

When, in place of the 208.5 parts of N-(2-hydroxyhexadecyl)-iminodiacetic acid, disodium salt, the same reaction is effected, using a stoichiometrically equivalent amount of N-(2-hydroxy-n-butyl)-iminodipropionic acid, potassium salt; N-(2-hydroxy-6-ethyl-n-hexadecyl)-iminoditoluic acid, ammonium salt; N-(2-hydroxy-N-decyl-imino-isopropyl-benzoic acid, triethanolamine salt; or N-(2-hydroxyoctadecyl)-imino-acetic-propionic acid, magnesium salt; the corresponding N-oxides are made. In some cases, to improve solubility of the reactants or the product in the reaction mixture, additional water is added or a suitable solvent, e.g., ethanol, n-butanol, isopropanol, is added after completion of the reaction, when the H₂O₂ has been destroyed, to improve the fluidity of the product.

EXAMPLE 3

Fifty parts of N-(2-hydroxydodecyl)-N-methyl glycine, sodium salt, are dissolved in 150 parts water. 22 parts of a 30 percent aqueous solution of hydrogen peroxide are added slowly at room temperature. A slight temperature increase is noted. After standing for 5 hours, the temperature is increased to 80°C. and held at this temperature for a period of 1 to 8 hours, until all excess hydrogen peroxide is decomposed. The product resulting is a liquid containing approximately 25 percent of the desired amine oxide. The solution is clear and is employed as is or is dried by a suitable technique, e.g., freeze drying, flash vaporization, evaporation, or oven drying, to produce a white hygroscopic powder.

When, in place of the N-(2-hydroxydodecyl)-N-methyl glycine, sodium salt, there are used 50 parts of N-(2-hydroxytetradecyl)-N-methyl glycine, sodium salt, the corresponding N-(2-hydroxytetradecyl)-N-

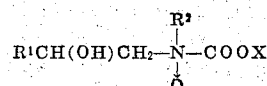
methyl glycine-N-oxide, sodium salt is made. The properties of this compounds are very similar to those of the corresponding N-(2-hydroxydodecyl)- compound.

When 50 parts of N-(2-hydroxyhexadecyl)-N-methyl glycine, sodium salt are used instead, the reaction mixture becomes gel-like and stringy before being heated to 80°C. After such heating, the 25 percent active ingredient product resulting is an opaque, viscous solution, rather than the thinner, clear solution previously obtained.

Corresponding products result when in place of the sodium salts described above, in this example, the salts used are those of potassium, triethanolamine, triisopropylamine, di-n-propylamine, trimethylamine, magnesium, ammonium, or potassium. Also, in these reactions the hydrogen peroxide may be removed by treatment with sodium sulfite, with just enough sodium sulfite being added to reduce the excess of hydrogen peroxide. Of course, such reducing agent would not be employed if the presence of sodium sulfate was objectionable in the product. However, this chemical decomposition of excess hydrogen peroxide appears to give a solution of improved color, as compared to the light yellow solution sometimes obtained when the peroxide is removed by thermal decomposition, as previously described. In these examples, the long chain alkyl groups of the hydroxy-alkyl are straight chain and terminally joined to the nitrogen, unless otherwise indicated.

EXAMPLE 4

Although general utilities as surface active agents, surface tension reducing agents, emulsifiers, detergents or softening agents for textiles have been noted for the products of Examples 1-3, especially useful softening and deterative actions have been observed for those compounds of the general formula



wherein R¹ is of 10 to 14 carbon atoms. Illustrative of such properties of these compounds are the data given in Tables 1 and 2, below.

TABLE 1

| Softening Effects | Softness |
|---|----------|
| N-(2-hydroxydodecyl)-N-methyl glycine-N-oxide, sodium salt | 10 |
| N-(2-hydroxytetradecyl)-N-methyl glycine-N-oxide, sodium salt | 10 |
| N-(2-hydroxyhexadecyl)-N-methyl glycine-N-oxide, sodium salt | 10 + |
| N-(2-hydroxyhexadecyl) iminodiacetic acid-N-oxide, di-sodium salt | 4 |

TABLE 2

Spangler Soil Detergency Tests

| | ΔRd (Soil Removal) | | | |
|---|--------------------|------------|----------|------------|
| | 120°F. | | 70°F. | |
| | N.B. Tap | 300 p.p.m. | N.B. Tap | 300 p.p.m. |
| N-(2-hydroxydodecyl)-N-methyl glycine-N-oxide, sodium salt | 14.9 | 10.0 | 12.5 | 10.7 |
| N-(2-hydroxytetradecyl)-N-methyl glycine-N-oxide, sodium salt | 16.0 | 11.4 | 14.2 | 12.1 |

N-(2-hydroxyhexadecyl)-N-methylglycine-N-oxide, sodium salt
N-(2-hydroxyhexadecyl) iminodiacetic acid-N-oxide, di-sodium salt

| | | | |
|------|------|------|------|
| 16.1 | 12.7 | 12.9 | 11.1 |
| 14.3 | 10.6 | 16.7 | 10.4 |

The softening effect test is one in which two grams of detergent material, made according to the methods of either Example 1 or Example 2, are blended with 6.6 grams of sodium tripolyphosphate and are used to wash one-half of a terrycloth hand towel, in three gallons of New Brunswick, New Jersey tap water (90 p.p.m. hardness), at 120°F. The washing is carried out, using a General Electric washing machine with a Mini-basket attachment in the machine. In a similar manner, controlled softness formulations are employed and towels washed therewith are compared to those washed, using the experimental softening compounds. The towels employed are of measurement 13 inches by 16 inches, purchased from J. C. Penney Company as 16 inch by 26 inch towels, which are cut in half for the present testing. Before testing, new towels are washed, two dozen towels per wash load, in a standard General Electric automatic washer, not using the Mini-basket, and employing a full amount of water in which are dissolved 10 grams of linear tridecyl benzene sulfonate active ingredient and 40 grams of sodium tripolyphosphate. This operation removes any finish applied at the mill, which might otherwise make the result of the test inaccurate.

The softness of the towels washed by the various compositions is rated on the scale of 1 to 10, with 1 indicating a towel that is not soft and 10 indicating excellent softness. Such ratings are made by comparison with control towels washed in detergents or softening agents of known characteristics.

The Spangler soil detergency tests are run using three cotton percale swatches, each 3 inches by 6 inches, each soiled with Spangler soil (a mixture of airborne and sebum soils). Washing of the swatches is done in a Tergotometer evaluating washing machine. The washing is done at two hardnesses and two temperatures, as indicated. The washing composition comprises 15 percent of each of the mentioned experimental compounds, 35 percent sodium tripolyphosphate and 50 percent sodium sulfate. The concentration of the washing preparation employed is 0.15 percent, which corresponds to the recommended usage of such materials in washing machines. Swatches are tested with a color difference meter before and after washing, using the Rd scale. The difference in Rd or delta Rd is

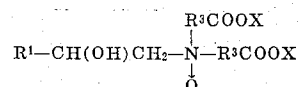
calculated and is reported. Obviously, the greater the delta Rd, the more efficient the removal of soil and the better detergency obtained. Linear tridecyl benzene sulfonate, sodium salt, is usually employed as a standard of comparison for detergency, in this test.

From the data, it is apparent that the novel compounds of this invention described herein are useful detergents. Comparative tests of detergency, against a standard linear tridecyl benzene sulfonate detergent, show that comparable detergency is obtained in hard water at a 120°F. washing temperature and a superior detergency is obtained in cold hard water, at 70°F. In cold water washing, using New Brunswick, New Jersey tap water, detergency in some cases is similar to the control, whereas at 120°F. the control is better.

The invention has been described and various illustrations and embodiments thereof have been set forth in the preceding specification. However, it is evident that equivalents may be substituted for the present compounds and procedural steps, without departing from the principles of this invention or the spirit thereof. Those of skill in the art will recognize that such equivalents are also within the present disclosure.

What is claimed is:

1. Compounds of the formula



wherein R¹ is alkyl or alkenyl of four to 20 carbon atoms, said alkenyl containing one or two double bonds, R³ is an alkylene or an aromatic hydrocarbon radical of one to nine carbon atoms and X is hydrogen, alkali metal, alkaline earth metal, ammonium, monoalkylamine, dialkylamine, trialkylamine, monoalkanolamine, dialkanolamine or trialkanolamine, in which the alkyl and alkanol groups are of one to four carbon atoms.

2. Compounds according to claim 1 wherein X is monovalent.

3. Compounds according to claim 2 wherein R¹ is of eight to 14 carbon atoms, R³ is alkylene of 1 to 3 carbon atoms, both R³'s are the same and both X's are the same.

4. Compounds according to claim 3 wherein R¹ is a linear alkyl radical of 10 to 14 carbon atoms, R³ is methylene and X is alkali metal.

5. A compound according to claim 4 wherein R¹ is linear alkyl of 14 carbon atoms, terminally joined to the rest of the compound, and X is sodium.

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