

ANTISTATIC COMPOSITIONS CONTAINING N-HIGHER
ALK(EN)YL NEOALKANOAMIDES

The present invention relates to alkanoamides which have been found to be adsorbable from wash and rinse waters by fibrous materials, such as the fabrics of items of household laundry, especially synthetic polymeric fibres thereof, such as polyesters, and which have been found to impart to such fabrics antistatic properties, so that the accumulation or the development of electrostatic charges thereon is inhibited. The present invention relates to antistatic compositions containing N-higher alkyl and alkenyl neoalkanoamides of 5 to 16 carbon atoms in the acyl moieties thereof and 8 to 20 carbon atoms in the alkyl and/or alkenyl groups of the amine moieties thereof. The term higher alk(en)yl used herein means a group of 8 to 20 carbon atoms. Examples of such compounds are neodecanoamides, neopentanoamides, neoheptanoamides, neononanoamides, neododecanoamides, neotridecanoamides and neotetradecanoamides. The invention also extends to methods for their manufacture; detergent, rinse and other antistatic compositions and products containing them; and methods of treating laundry with such compositions in washing, rinsing and other operations to impart antistatic properties to it.

Neodecanoic and neopentanoic acids are presently being marketed by Exxon Chemical Americas and are described in a bulletin issued by such company and entitled Neo Acids Properties, Chemistry and Applications (copyright 1982). Other neoalkanoic acids have also been made, such as neoheptanoic acids, neononanoic acid and mixed neododecanoic, neotridecanoic and neotetradecanoic acids. Amides of neo acids and methods for their manufacture are referred to broadly at page 10, column 1 of such

bulletin, and uses of various neodalkanoamides including neodecanoamide are mentioned therein, including applications as pesticides, plasticisers (for polyvinyl chloride), foam boosters, foam
5 suppressants, and slip agents (for polyolefin films). However, no mention is made of any of the neoalkanolamides of the present invention and the preferred manufacturing method, which results in the production of a light coloured better product, is not
10 taught, nor are they suggested for use as antistatic agents.

Computer searches of U.S. patents for the period 1950-1984 and of Chemical Abstracts for the period 1967-1985 have resulted in the finding of U.S. patent
15 4,440,666, directed to a hydrocarbon liquid containing a minor proportion of a reaction product of a polyalkylene polyamine and a neo-acid of 5 to 20 carbon atoms, in which the amide acts as a corrosion inhibitor. This patent does not appear to disclose
20 any N-higher alkyl or alkenyl neoalkanoamide and does not suggested that any such compounds would have antistatic properties. None of the other references found in the computer search discloses or suggests N-higher alkyl or alkenyl neoalkanoamides, any
25 antistatic properties of such or closely related compounds, or their inclusion in detergent, rinse or other laundry treating compositions, and none discloses or suggests applicants' process for manufacturing such amides of better colour (and
30 higher purity).

Because modern synthetic organic detergents are

excellent cleaning agents and, unlike soaps, do not normally leave deposits of fatty materials on washed laundry, often laundry washed with them lacks desirable softness. Because synthetic polymeric

5. fibres, which are the fibres of the fabrics of much of such laundry, are susceptible to disadvantageous accumulations of static charges, which occur during machine drying or when the fabrics are rubbed against other materials, with such tendency to static
10. accumulation and resulting discharging or sparking being exacerbated by the absence of any fatty acid or insoluble soap coating on the fibres, much research has been conducted in an effort to discover materials which, when incorporated in detergent compositions or
15. in rinse waters, would diminish any static charges on the laundry or would inhibit the accumulation of such charges.

- Quaternary ammonium salts, such as di-lower alkyl di-higher alkyl ammonium halides, e.g. dimethyl
20. distearyl ammonium chloride, have been used as fabric softeners in detergents, in fabric softening compositions for addition to the rinse water, and in papers, sponges and other substrates intended for introduction into laundry dryers, where they transfer
 25. such cationic materials to the laundry being tumbled. Certain amines have also been found to be useful in some such applications. However, because such cationic materials react objectionably with anionic detergents their use in anionic detergent
 30. compositions results in production of undesirable

reaction products and causes losses of detergency.

- It is an important feature of the present invention that certain amides have been discovered which are water insoluble and which may be in desired
5. oily or plastic, flowable or spreadable state at normal use temperatures, e.g. 10° to 90°C, preferably 10° to 60°C. Particular amides which have the desired physical characteristics are adsorbable or otherwise depositable onto laundry from the wash
10. water or rinse water in a washing machine, or can be deposited onto drying laundry in the dryer, are higher amides of neoalkanoic acids. In accordance with the present invention such novel compounds are N-higher alkyl and alkenyl neoalkanoamides or
15. neoalkanoic acids, such as amides previously mentioned, the acid moieties of which have 5 to 16 carbon atoms and preferably contain from 7 to 14 carbon atoms. Although some branching of the hydrocarbyls is acceptable under certain
20. circumstances, it is preferable that the alkyl and alkenyl groups be substantially or essentially linear, and more preferably, they will be linear. Among the more preferable of the neoalkanoamides are those wherein the alkyl or alkenyl is higher, of 8 to
25. 20 carbon atoms, often preferably 12 to 18 carbon atoms, such as may be derived from coconut oil, tallow or hydrogenated tallow, which for the higher alkyls are herein referred to as coco alkyl, tallow alkyl and hydrogenated tallow alkyl, respectively.
30. It should be noted that in this usage "alkyl" may be

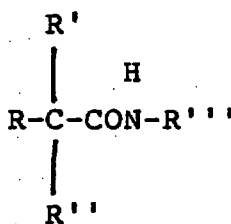
- inclusive of hydrocarbyl groups containing minor unsaturation, as in tallow alkyl, which contains a minor proportion of a monounsaturated $C_{18}H_{35}$ group. Also within the invention are: a process for the
5. production of such N-higher alk(en)yl neoalkanoamides; detergent composition such a neoalkanoamide and a fabric softening proportion of bentonite; rinse compositions containing both such constituents; a substrate material containing such a neoalkanoamide,
 10. for use in a laundry dryer; and processes for treating laundry with the neoalkanoamide during washing, rinsing and/or drying.

- Neodecanoic acid, which is available commercially from Exxon Chemical Americas in prime and technical grades, is synthesised by reacting a
15. branched nonene and carbon monoxide under high pressure at an elevated temperature in the presence of an aqueous acidic catalyst (Koch reaction). The general mechanism involved includes generation of a
 20. carbonium ion followed by complexation with carbon monoxide and the catalyst to form a "complex", which is subsequently hydrolysed to generate the free acid. The formula of the free acid is:



wherein the number of carbon atoms is $R + R' + R''$ is 8; about 31% of the neodecanoic acid is of a structure wherein R' and R'' are both methyl and R is hexyl; 67% is of a formula wherein R' is methyl, R'' is of a carbon atoms content greater than that of methyl and less than that of R , and R is of a carbon atoms content less than that of hexyl and greater than that of R'' ; and 2% is of a formula wherein R' and R'' are both of a carbon atoms content greater than that of methyl and less than that of R , and R is of a carbon atoms content less than that of hexyl and greater than those of R' and R'' . The dissociation constant (K_a) of neodecanoic acid is 4.20×10^{-6} . Among other neoalkanoic acids that are available may be mentioned others in the 5 to 16 carbon atom content range, such as neopentanoic, neoheptanoic, neononanoic, neodecanoic, neododecanoic, neotridecanoic and neotetradecanoic acids.

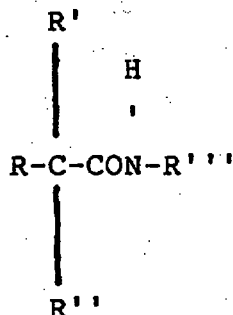
According to the first aspect of the present invention there are provided compositions containing antistatic N-higher alk(en)yl neoalkanoamides having the formula



in which R , R' and R'' each represent alkyl groups which may be the same or different and in which the sum of the carbon atoms in the groups R , R' and R'' is 3 to 14 and R''' represents an alkyl or alkenyl group having 8 to 20 carbon atoms.

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According to a second aspect of the present invention there are provided N-higher alk(en)yl neoalkanoamides having the formula

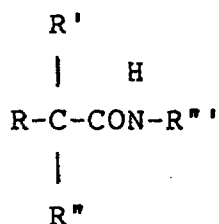


in which R, R' and R'' each represent alkyl groups which may be the same or different and in which the sum of the carbon atoms in the groups R, R' and R'' is 3 to 14 and R''' represents an alkyl or alkenyl group having 8 to 20 carbon atoms, the R''' groups being linear.

To make the neoalkanoamides useful in this invention the neoalkanoic acid, such as neodecanoic acid, may be reacted directly with a higher alkyl- or alkenyl-amine, which is very preferably a linear primary amine, R'''NH₂, but also may include slightly branched alkyls having less than 10 or 20% of their carbon atoms contents in branch(es), e.g. as in 2-methyl heptadecane. The higher alkylamines and alkenylamines employed will be of a number of carbon atoms in the range of 8 to 20, often preferably 12 to 18.

Among the more preferred of the amine starting materials are coco alkyl amine, tallow alkyl amine (which contains a minor portion of oleyl amine) and hydrogenated tallow alkyl amine. Such materials are available from vegetable and animal sources, and amides made from them have been found to be excellent antistats, which are compatible with anionic detergents. Also especially useful amine starting materials are oleyl amine and octyl amine.

The amides useful in and of the present invention, which are of the formula:



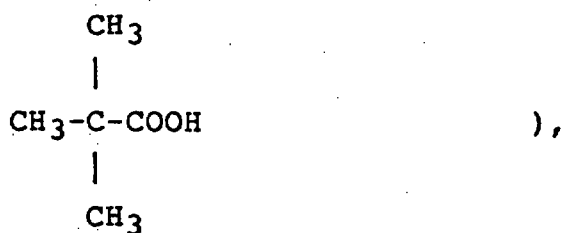
for the neoalkanoamides, may be made by reacting a neoalkanoyl chloride with a higher alkyl or alkenyl amine, $\text{R}''\text{NH}_2$, but a less costly synthesis is directly from the neoalkanoic acid by reacting it with such amine at an elevated temperature. The product of such reaction, unfortunately, is often very dark in colour, which can make it unsuitable for incorporation in desirably white detergent compositions, which would be discoloured by the presence of the amide. Applicants have found that by reacting the neoalkanoic acid with the higher alkyl or alkenyl

amine, e.g., coco alkyl amine, at a suitable elevated temperature, preferably about 250°C, for the coco-neodecanoamide, under an inert gas or nitrogen atmosphere, a nearly water white oily product result (the desired amide) which constitutes a first, upper phase, and may be readily separated from a second, lower phase, which includes by-products and any excess of reactants.

Normally, the reaction temperature for making the various neoalkanoamides useful in and of the present invention will be in the range of 180 to 320°C and reaction times will be from 5 to 8 hours, with stirring being continued during the entire reaction. The melting points of the products will normally be low, so that the products will desirably be liquids at room temperature or at normal use temperatures. The melting points of the coco alkyl-tallow alkyl- and hydrogenated tallow alkyl-neodecanoamides are 0°C, 15 to 17°C and 45 to 49°C, respectively, while those of the octyl, oleyl, palmityl and stearyl counterparts are less than 0°C, 5 to 6°C, 37 to 38°C and 35 to 40°C, respectively. The refractive indices for the coco alkyl and octyl neodecanoamides are 1.4626 and 1.4596, respectively. Melting points for the other neoalkanoamides of 5 to 16 carbon atoms in the neoalkanoic acid will be in the less than 0° to 60°C range and preferably the amides will be oily liquids at temperatures of 50°C or less, and may be in solid state at about room temperature.

Although the described N-higher alkyl neodecano-

amides are the preferred embodiments of the present invention, other highly branched acids may also be employed for the manufacture of higher alkyl amide antistats. When neopentanoic acid is employed (it is of the formula



for the manufacture of N-higher alkyl neopentano-amides antistatic action is obtained but not to the extent realised for the higher alkyl neodecanoamides. Normally the neoacid employed will be of 5 to 16, preferably 7 to 14 carbon atoms, and such acids are obtainable by the described process when highly branched C₄-C₁₅ or C₆-C₁₃ olefins are employed as starting materials in the Koch reaction.

Antistatic N-higher alk(en)yl neoalkanoamides of the present invention may be employed to treat various fibrous materials, including polyesters, nylons, polyacrylates and acetates, blends of some or all of such materials and blends of any of such materials with natural fibres, such as cotton, to lower the tendencies thereof to accumulate objectionable static charges. They may also be used to treat non-fibrous polymeric materials, such as video tapes and cassettes, camera films and photographs, movie film,

sound recording tapes and cassettes, plastic sheets and moulded (and otherwise formed) plastic items, such as items made from polyvinyl chloride (or polyvinyl chloride sheeting). In such treatments, the amides may be applied directly or in suspension or solution, as liquids, pastes or sprays, to the surfaces of the items to be treated, in comparatively small proportions, normally with the proportion of amide to that of treated materials being in the range of 0.0001 to 0.2%, by weight.

Although the antistatic agents of the present invention may be applied directly or in suspension or solution to materials being treated to make them static-free it is usually highly preferable for them to be incorporated in other compositions that are used for different treatment for such materials. Thus, it is desirable for such antistatic agents to be incorporable in detergent compositions so that the laundry washed with such compositions will not accumulate annoying static charges. Such compositions will comprise a deterative proportion of a synthetic organic detergent and a sufficient proportion of an N-higher alkyl neoalkanoamide to impart antistatic characteristics to washed laundry.

The neoalkanoamides useful in and of the present invention are especially advantageous for use in detergent compositions of the anionic type because, unlike quaternary ammonium halides, they do not react adversely with anionic detergents. Thus, they do not form objectionable fatty reaction products which can

- deposit on and mar the appearance of washed laundry, and they do not cause a diminution in deterative activity of the detergent composition. Furthermore, they are effective antistatic agents, being
5. adsorbable onto washed laundry, especially synthetic polymeric fibres thereof, during the washing process. In preferred detergent compositions of the present invention the synthetic organic detergent will be of the sulphate and/or sulphonate type, normally
 10. including a higher aliphatic chain, such as a higher alkyl of 8 to 20 carbon atoms, in the lipophilic portion thereof. Preferably, such materials will be employed as water soluble salts, e.g. sodium salts. While the present neoalkanoamides may be employed in
 15. nonionic detergent compositions or detergent compositions of various types, including amphoteric, ampholytic or zwitterionic detergents, preferably the detergent will be an anionic detergent and will usually be one or more of the following: linear
 20. higher alkylbenzene sulphonates; branched higher alkylbenzene sulphonates; higher fatty alcohol sulphates; olefin sulphonates; paraffin sulphonates; monoglyceride sulphates; fatty alcohol ethoxylate sulphates; higher fatty acid sulfoesters of
 25. isethionic acid; higher fatty acyl sarcosides; and acyl- and sulfo-amides of N-methyl taurine. In such detergents there will normally be present a higher aliphatic or alkyl group, which is preferably linear, and which will usually be of 8 to 20 carbon atoms,
 30. more preferably 12 to 18. While lower alkoxy chains are present, as in the mentioned ethoxylate sulphate,

usually there will be from 3 to 30 ethoxy groups, preferably 3 to 10. Such detergents will normally be employed as sodium salts, although other water soluble salts, such as potassium, ammonium and triethanolamine salts, may be used too, in certain circumstances.

For heavy duty laundering applications the detergent compositions will usually contain a builder, to increase the detergency of the anionic detergent, especially in hard water. Among the various builders that may be utilised those of preference include: the polyphosphates, sodium tripolyphosphate and tetrasodium pyrophosphate; carbonates; bicarbonates; sesquicarbonates; silicates; sesquisilicates; citrates; nitrilotriacetates; and polyacetal carboxylates; all of which are water soluble salts, and the water softening zeolites, such as hydrated Zeolite A, which are water insoluble.

The proportion of the neoalkanoamide in the detergent composition will be an antistatic characteristic imparting proportion (to washed laundry adsorbing the neoalkanoamide during washing) and such will normally be in the range of about 1 to 20% of the detergent composition, by weight, preferably being 3 to 15% and more preferably being 5 to 12%, e.g. about 10%. When heavy loads of laundry are to be treated the content of neoalkanolamide in the detergent composition will preferably be about 6 to 10% or more, often 7 to 10%.

- In addition to the neoalkanoamide, detergent and builder, the detergent compositions of the present invention, even when in solid or particulate form, will also usually contain some moisture. The
5. proportion of moisture will usually be in the range of 2 to 20%, preferably being 3 to 15% and more preferably about 5 to 12%, e.g. about 8%. The particulate material will desirably be in the form of spray dried detergent composition beads, with
 10. particle sizes in the range of Nos. 10 to 140 (which have openings 200 to 105 microns across), preferably 10 to 100, (which have openings 200 to 149 microns across), U.S. Sieve Series. Other forms of the detergent composition may be made, including liquids,
 15. gels, pastes, bars and cakes, and the particulate compositions and the compositions in such other forms will also normally contain functional and aesthetic adjuvants, and may contain fillers. Such adjuvants and fillers will normally comprise the balance of the
 20. detergents. Among the adjuvants that may be employed are: fluorescent or optical brightening agents, such as stilbene brighteners; anti-redeposition agents, such as sodium carboxymethyl cellulose; soil release promoting polymers, such as Alkaril QCF; fabric
 25. softening agents, such as bentonite; anti-gelling agents (for use in the crutcher), such as citric acid and magnesium sulphate; colourants, such as ultramarine blue pigment and dyes; whitening agents, such as titanium dioxide; enzymes, such as mixed
 30. proteolytic and amylolytic enzymes; and perfumes.

- Among the fillers or bodying agents that are sometimes employed the most preferred is sodium sulphate, although sodium chloride has also been used. In liquid detergent compositions water, lower alcohols, glycols, co-solvents, and anti-freeze additives may also be present.

- The proportions of detergent, builder, N-higher alkyl neoalkanoamide and moisture in the particulate antistatic detergents of the present invention will normally be within the ranges of 5 to 35%, 10 to 85%, 1 to 20% and 2 to 20%, respectively. Preferred proportions are 8 to 30%, 25 to 70%, 3 to 15% and 3 to 15%, respectively, with more preferred proportions being 10 to 25%, 30 to 70%, 5 to 12% and 5 to 12%, respectively. The moisture content includes hydrate moisture that is removed by the standard moisture test, namely heating for one hour at 105°C, and such removed moisture is not included in the weights of the other components of the composition.
- The detergent composition, when in particulate form, may be made by spray drying an aqueous crutcher mix of the various constituents thereof to free flowing bead form, utilising well known spray drying equipment and following a standard spray drying procedure, in which a hot drying gas, which is usually the products of combustion of fuel oil or gas, passes concurrently or countercurrently, with respect to falling spray droplets of an aqueous crutcher mix, to produce the dried beads, which are removed from the bottom of the spray tower, and subsequently may be screened or otherwise classified to desired

particle size range. The resulting beads are excellent detergents and are capable of having the antistat component thereof adsorbed by laundry to lower any tendency of laundry to accumulate static charges. However, even greater antistatic activity is observable when the neoalkanoamide is not spray dried with the rest of the detergent composition but is sprayed onto or otherwise applied to the spray dried detergent composition particles, base beads or to a detergent composition made by mixing together particulate components thereof. In a preferred process the neoalkanoamide is dissolved in the formula proportion of liquefiable nonionic detergent at elevated temperature (40°-50°C) and the solution is sprayed onto and absorbed by porous spray dried builder beads. The mentioned improved antistatic results are also obtainable by addition of the neoalkanoamide antistat to the wash water, with separate addition thereto of the detergent composition. For such and other uses the antistat may be made in a convenient powder form for use by being first mixed with a suitable carrier, such as Microcel (a synthetic calcium silicate powder), a filler, e.g. particulate sodium sulphate, or a softening agent, e.g. bentonite, or other suitable material. When liquid, gel or paste detergent compositions are made, wherein the proportion of solvent or liquid medium is different from the moisture content of the solid or particulate products, the proportions of detergent, antistat, builder, when present, and

adjuvants, when present, will be adjusted accordingly, normally with the relative proportions thereof being maintained about the same as in the solid compositions. However, the proportion of the neoalkanoamide antistat in such detergent compositions and in other antistatic preparations will be maintained such as to be capable of imparting antistatic properties to the material to be treated, when the composition is employed in appropriate manner. One of skill in the art will be able to modify the formulations so as to make products of greatest utility and satisfactory stability. Similarly, it is contemplated that the formula will be changed when it is desired to produce compositions useful in the rinse or in the dryer. Rinse compositions may sometimes contain only the neoalkanoamide dissolved in a suitable solvent medium or dispersed in an aqueous liquid medium, preferably with the aid of a hydrotrope or other surface active component. The proportion of antistat will preferably be kept about the same as that for the antistatic detergent composition previously discussed, e.g. 5 to 12%, although less could be used because in the absence of the detergent and builder the antistat will usually be more substantive. For liquid preparations for use in the rinse water, the proportion of solvent or liquid will normally be from 30 to 90%, while any surface active material or hydrotrope content will usually be in the range of 0.1 to 5%. If quaternary ammonium halide is

also present the proportion thereof will desirably be in the range of one part of the quaternary compound to 1/2 to 10 parts of neoalkanoamide antistat.

5 Additionally, when a polyurethane or cellulose sponge strip or a textile paper substrate is impregnated with the antistat of the present invention (usually with the weight percentage thereof being from 10 to 100% of the weight of the substrate), a fatty material, such as monoglyceride or diglyceride of 10 higher fatty acids may be present too, to aid in the depositing of the neoalkanoamide onto the surfaces of the fabric fibres. A suitable such material is coconut oil fatty acids diglyceride.

15 When the neoalkanoamide antistatic agent of the present invention is applied to laundry during the washing or rinsing operations, by adsorption thereof onto the laundry in the wash water or the rinse, the concentration of the detergent composition or the 20 rinse preparation in the wash water will be sufficient to impart antistatic properties to the washed laundry, e.g. laundry items of polyester or polyester cotton blend fabrics. Such an effective concentration will normally be in the range of 0.005 to 0.1%, e.g. for N-higher alkyl neoalkanoamide and 25 preferably such range will be 0.01 to 0.05%. The detergent composition or rinse composition concentration in the wash water will normally be in the range of 0.05 to 0.5%, preferably being 0.08 to 0.2%. The wash or rinse water will normally be at a 30 temperature in the range of 10° to 90°C, e.g. 30° to

- 50°C, with the lower part of the 10° to 90°C washing temperature range being typical of American home laundry practice and the upper part of that range being that employed in European practice, especially when perborate-containing detergent compositions are employed (rinse temperature will normally be in the lower part of such range for both). In American practice the normal washing temperature will be in the range of 20° to 60°C and for "cold water washing" and rinsings such range is often from 20° to 40°C (or lower for rinsing). The washing operation will normally take between five minutes and one hour, with rinsing taking from two minutes to twenty minutes of that time. The water employed may be soft or hard and hardnesses between 0 and 250 ppm (mixed calcium and magnesium hardnesses, as calcium carbonate) may be encountered. Under such washing an/or rinsing conditions the neoalkanoamides of the present invention are sufficiently substantive to the laundry being washed, especially that of synthetic organic polymers, such as polyesters, to be adsorbed thereon in sufficient proportion to make the polymer antistatic, thereby diminishing any static charges that could otherwise accumulate on the polymer during a machine drying (tumbling) operation or as a result of frictional forces applied to the polymer surface, as by rubbing against other materials. When washed laundry is treated in the dryer with substrate materials onto which the neoalkanoamide of the present invention or a mixture thereof with
- 5.
- 10.
- 15.
- 20.
- 25.
- 30.

quaternary ammonium salt has been deposited it is found that the dried laundry resulting is of a diminished tendency to accumulate static charges.

While any of the methods of application of the neoalkanoamides to material to be treated may be employed, and good antistatic properties will be transmitted to the treated material, it is within the invention to utilise a plurality of such application operations, such as washing, rinsing and drying, some or all in the presence of the antistatic agent. Also, laundry may be brushed or sprayed with the antistatic agent in solution or dispersion, and other materials, such as carpeting, may be similarly treated. However, a significant advantage of the products of the present invention is in their compatibility with anionic detergents in detergent compositions and wash waters, wherein antistatic proportions of quaternary ammonium salts often have undesirable effects on the detergative action of the anionic detergents and cause objectionable reactions which often result in spotting with the reaction products of the laundry or other items being washed.

The invention may be put into practice in various ways and a number of specific embodiments will be described to illustrate the invention with reference to the accompanying examples and drawings which are infrared absorption spectra for several representative N-higher alk(en)yl neoalkanoamides and in which:-

Figure 1 is an infrared absorption spectrum for N-tallow alkyl neodecanoamide;

Figure 2 is an infrared absorption spectrum for N-coco alkyl neodecanoamide;

5. Figure 3 is an infrared absorption spectrum for N-hexadecyl neodecanoamide; and

Figure 4 is an infrared absorption spectrum for N-oleyl neodecanoamide.

Unless otherwise indicated, in these examples,
10. the specification and claims, all parts given are by weight and all temperatures are in °C.

EXAMPLES 1A to 1C

Example 1A

181 grams of Arman CD coco alkyl amine are
15. reacted with 142 grams of neodecanoic acid (prime grade, 95.2% pure) in a one-litre, 3-necked glass flask fitted with a magnetic stirrer, a heating mantle, an ice condenser, a nitrogen inlet and a communicating nitrogen source (to blanket the
20. reaction). Prior to the reaction the flask had been purged of air and a nitrogen atmosphere had been introduced, which was maintained during the atmospheric pressure reaction. The reaction was conducted at 300°C (the reaction range is from 180 to
25. 320°C for this and the other condensation reactions) and was monitored by observing the water collected from the condenser. After seven hours it was considered that the reaction was essentially complete (8 ml of water had been collected), and the flask was
30. removed from the heating mantle. After being allowed

- to stand at room temperature over a long weekend (about 90 hours) the contents were transferred to a one-litre separatory funnel, washed sequentially with a) 50:44:6 water:ethanol:HCl solution, b) 56:44
5. water:ethanol solution, c) 5% aqueous NaOH, and d) distilled water, until neutral. After completion of washing the excess water was drained off and the washed product was dried in a vacuum rotary evaporator, yielding 273 grams of product.
10. The product is a light coloured oil (Gardner No. 2), with a melting point of less than 0°C and a refractive index ($n_D^{20^\circ\text{C}}$) of 1.4626. The infrared absorption spectrum for the N-coco alkyl neodecanoamide made is given in Figure 1. It will be noted
15. that at about $3,350\text{ cm}^{-1}$ and $1,633\text{ cm}^{-1}$ strong absorption bands are present, indicating the presence in the compound of a secondary amide stretch (N-H) and a secondary amide carbonyl (C=O), respectively, and at 720 cm^{-1} there is a weak absorption band,
20. indicating the presence of a long alkyl chain. The nuclear magnetic resonance spectrum was obtained and was found to be consistent with the expected structure. A peak in the proton spectrum for NH appears at 5.7 ppm.
25. Example 1B
- When, instead of employing a nitrogen atmosphere over the reaction mixture there are used instead carbon dioxide, argon, or other gas inert to the reaction, or when vacuum is employed (preferably
30. lower than 25 cm of mercury), good, light coloured

product of essentially the same physical characteristics as mentioned above results, in similar good yield. When vacuum is utilised it may often be desirable to lower the reaction temperature accordingly (usually about 10-30°C) to prevent possible losses of reactants and/or product. When a suitable inert gaseous atmosphere is not provided above the reactants, as when air is the gas that is present, the N-coco alkyl neodecanoamide made is darker in colour and may be unsuitable for incorporation in a retail detergent composition intended to impart antistatic properties to washed laundry.

Example 1C

An alternative reaction for the production of N-higher coco alkyl neodecanoamide is the reaction of a gram-mole of a neodecanoyl chloride, which is slowly added over a period of about an hour to a gram-mole of coco alkyl amine dissolved in a solvent medium of 700 ml of diethyl ether and a gram-mole of triethyl amine (which acts as a trap for HCl). The coco alkyl amine is in a one-litre, 3-necked flask fitted with a condenser with a Drierite (Registered Trade Mark) tube, a thermometer, a Chesapeake stirrer and a dropping funnel, and the flask is cooled by an ice bath.

After completion of addition of the neodecanoyl chloride the ice bath is removed and the reaction mix is allowed to come to room temperature, after which it is stirred for an additional hour. It is then transferred to a two-litre separatory funnel and is

- washed twice with water, once with 5% aqueous hydrochloric acid and once with 5% aqueous sodium hydroxide, followed by one or more washings with distilled water until the product is neutral to pH
5. paper. Any remaining ether is removed by means of a steam bath and the product is finished on a vacuum rotary evaporator. The product made is water white to light amber in colour, is pure, and exhibits the infrared and NMR spectra previously described for the
10. same product made by the condensation method.

EXAMPLES 2A and 2B

Example 2A

- Essentially the same procedure as that described in Example 1A is followed, but the reactants are
15. Armak tallow amine (199 g) and neodecanoic acid (121 g, prime grade, 95.2% pure). The reaction was conducted under nitrogen over a period of about eight hours at a temperature in the range of 240-260°C and during that time 8 ml of water were collected. The
20. washing solutions employed were the same as in Example 1 but four "final" washes with distilled water were made to produce a tallow alkyl neodecanoamide that is neutral to pH paper. The final traces of water and alcohol were removed, utilising a rotary
25. evaporator. The yield was 159 grams of a light coloured product (Gardner colour = 2) which included 2.5% of the starting amine and 0.3% of the starting acid. The tallow alkyl neodecanoamide made has a melting point of 15 to 17°C and the infrared spectrum
30. for it is illustrated in Figure 2.

Example 2B

As was described in Example 1C for the coco alkyl neodecanoamide, the tallow alkyl neodecanoamide can also be made by the acid chloride process,

5. utilising equal molar proportions of neodecanoic acid and tallow alkyl amine.

- While the described condensation reaction, utilising heating of the reactants in a flask under inert atmosphere, usually takes from 5 to 8 hours at a temperature in the range of 180 to 320°C for the condensation process of this example and others herein given, longer reaction times, usually lower temperatures, may be employed and shorter reaction times, sometimes at higher temperatures, may be utilised. A manufacturing apparatus for such quicker reactions may be a thin film reactor or comparable equipment.

EXAMPLE 3

- N-tallow alkyl neopentanoamide is made by reacting 51 grams of neopentanoic acid (obtained from Exxon Chemical Americas) with 134 grams of tallow amine-TD (obtained from Armak Chemical Company). The reaction is conducted in a 500 ml, 3-necked flask, equipped with a magnetic stirrer, a condenser with a Dean-Stark trap, and a nitrogen inlet, connected to a source of nitrogen. The flask was heated to 250°C and after five hours the heat was turned off and the flask was allowed to set overnight. The product was transferred to a heated separatory funnel and was sequentially washed with aqueous alcoholic

- hydrochloric acid (53% water, 44% ethanol and 3 HCl), water-alcohol mixture (53% water, 47% ethanol), aqueous alcoholic sodium hydroxide (53% water 44% ethanol and 3% sodium hydroxide) and distilled water
5. (four washes), until the product was neutral to pH paper. The product made is of a melting point of 38° to 39°C.

EXAMPLES 4A to 4I

- The condensation reaction described in Examples
10. 1 to 3 is also practised to make
- N-methyl neodecanoamide (Example 4A),
N-ethyl neodecanoamide (Example 4B),
N-t-butyl neodecanoamide (Example 4C),
N-octyl neodecanoamide (Example 4D),
15. N-myristyl neodecanoamide (Example 4E),
N-hexadecyl neodecanoamide (or N-palmityl neodecanoamide) (Example 4F),
N-oleyl neodecanoamide (Example 4G),
N-hydrogenated tallow neodecanoamide (Example
20. 4H), and
N-stearyl neodecanoamide (Example 4I).

- In all such reactions the apparatus employed is like that described in Examples 1 to 3, a nitrogen blanket is utilised, the time of reaction is from 5 to
25. 8 hours and the reaction temperature is a suitable temperature in the range of 180° to 300°C. For the normally solid products the washings are conducted in a heated separatory funnel and heated washing agents are utilised.
30. The N-octyl neodecanoamide (Example 4D) and the

N-oley1 neodecanoamide (Example 4G) are both oily materials, like the N-tallow alkyl neodecanoamide (Example 2) and N-coco alkyl neodecanoamide (Example 1), and exhibit greater adsorptions onto fabrics or fibrous materials than do the other low melting

5. neodecanoamides made, which allows them to function more satisfactorily as antistatic agents, useful for incorporation in detergent compositions to impart antistatic activity to washed laundry. The melting points of the products made are listed below in table
10. 1, together with the refractive indices for some that have melting points below 0°C.

TABLE 1

	Alk(en)yl (in N-alk(en)yl Neodecanoamide)	Melting Point (°C)	Refractive Index (n_D 20°C)
15.	Methyl	< 0	1.4554
	Ethyl	< 0	1.4554
	t-Butyl	< 0	
	Octyl	< 0	1.4596
20.	Myristyl	< 0	1.4612
	Palmityl	37-38	
	Oleyl	5-6	
	Stearyl	35-40	
	Hydrogenated tallow	45-49	

25.

In addition to varying the alk(en)yl group of the N-alk(en)yl neodecanoamides of the present invention within the 8 to 20 carbon range, as described in this example and in Examples 1 and 2,

30.

- the neoalkanoic acid moiety of the amides of the present invention may also be changed. Thus, the condensation and acid chloride reactions of this example and Examples 1 to 3 may be practised, with
5. the substitution for the neodecanoic acid of this example and Examples 1 and 2 and for the neopentanoic acid of Example 3, of other neoalkanoic acids of carbon atoms contents in the range of 5 to 16, specifically neoheptanoic acid, neononanoic acid,
 10. neododecanoic acid, neotridecanoic acid and neotetradecanoic acid. The products within this invention that are made by the described reactions, especially the condensation reaction, as conducted under an inert gas atmosphere and with acidic, basic
 15. and distilled water washings of the product to pH neutrality, are amides with antistatic properties, which make them useful for the treatment of laundry to decrease or prevent static clinging thereof after machine drying. Especially preferred are the oily
 20. appearing amides, such as those wherein the alk(en)yl groups is octyl, myristyl, oleyl or coco alkyl. However, the tallow alkyl neodecanoamides and neopentanoamides are also useful antistatic agents and additionally possess fabric softening properties,
 25. especially when employed in conjunction with bentonite.

EXAMPLE 5

A composition is made having the formulation given in Table 2 below.

Table 2

5.	<u>Component</u>	<u>Percent</u>
	Sodium linear tridecylbenzene sulphonate	13.4
	Sodium tripolyphosphate	24.0
	Sodium silicate ($\text{Na}_2\text{O}:\text{SiO}_2 = 1:2.4$)	6.3
	Sodium carbonate	4.5
10.	Borax	1.0
	Fluorescent brighteners	0.3
	Methyl cellulose	0.5
	Sodium carboxymethyl cellulose	0.2
	Sodium sulphate	49.6
15.	Perfume	0.2
		<hr/> 100.0

20. A spray dried detergent composition of the above formulation is made by spray drying an aqueous crutcher mix of 60% solids content in a conventional countercurrent spray drying tower to produce spray dried detergent beads, less perfume, which beads are subsequently perfumed by spraying onto the surfaces thereof the formula proportion of liquid perfume.
25. The product is screened so that the particle sizes thereof will be in the range of Nos. 10 to 100, U.S. Sieve Series (which have openings 200 to 149 microns across). Then, desired proportions of oily neodecano-
30. amides, and N-tallow alkyl neopentanoamide are

sprayed onto the detergent composition beads to produce antistatic detergent compositions. Instead of mixing the neoalkanoamide with the detergent composition to make an antistatic detergent composition, it may be added to the wash water, and sometimes is preferably added to the rinse. The effects of the antistat materials are evaluated by washing test fabrics in top loading Whirlpool (Registered Trade Mark) washing machines and drying them in electric automatic clothes dryers, after which they are tested for static accumulations. A ballast load is employed in the washing machine with the test swatches, which are also present to check on any possible negative effect of the antistat on soil removal properties of the detergent composition. The ballast load (5 lbs) consists of 1/3 cotton terry face cloths; 1/3 cotton percale swatches 14" x 15" (35.6 cm x 38.1 cm), and 1/3 of 65% Dacron (Registered Trade Mark): 35% cotton swatches 14" x 15" (35.6 cm x 38.1 cm) without durable press finish). The test swatches used for antistatic effect measurement are 14" x 15" (35.6 cm x 38.1 cm) and include one each of: Dacron double knit twill; 65% Dacron: 35% cotton permanent press; blue 65% Dacron: 35% cotton permanent press; Banlon (Registered Trade Mark); acetate jersey; and nylon tricot. The soil removal index swatches measured 3" x 6" (7.6 cm x 15.2 cm) and four of each type are present with the ballast laundry. The five different types of such swatches are Test Fabrics Inc. soil on nylon, Test Fabrics Inc. soil on cotton; Piscataway (New Jersey) clay on

cotton; Piscataway clay on 65% Dacron: 35% cotton cloth; and EMPA 101 oily soil on 65% Dacron: 35% cotton cloth.

- After thorough cleaning of the washers and
5. dryers, using 3A denatured alcohol, followed by air drying, the washing machine is set for a 14 minute wash time, using 17 gallons (65 litres) of water at 120°F (49°C). This "hot" wash is one utilising the normal machine cycle, including a cold rinse with tap
 10. water. The detergent composition, containing the antistat is added to the wash water after the machine is filled, the machine is allowed to agitate for about 10 seconds and then the ballast load and the various test and soil removal index swatches are
 15. added separately, while agitation is continued. Subsequently, the various fabrics are removed and placed in the electric dryer, where they are dried over a period of about two hours. The test swatches and two terry towels from the ballast are then dried
 20. for an additional 10 minutes and the test swatches are then evaluated for static cling. Prior to instrumental static measurements the test swatches are hung in a low humidity room (25% relative humidity) overnight. The Blue 65 Dacron: 35 cotton
 25. swatch may be examined for product spotting and the reflectance (Rd) values of the soil removal index swatches may be determined, utilising a reflectometer. Also, the cotton terry towels from the ballast may be evaluated for softness. To determine the static
 30. charges on the average test material, after washing

- with the detergent composition containing antistatic agent, all of the static test swatches are rubbed in a controlled manner, with wool, under controlled conditions, at a relative humidity in the range of 25
5. to 30%, after which the electrostatic charges on the swatches are measured and the measured electrostatic charges are averaged for each material, after which the averages for the materials are again averaged, resulting in a static index. It has been found that
10. differences of as little as 6 index units (in kilovolts) are significant and indicate that consumers will notice the difference in the static clings of washed materials different in static indices by six units.
15. The following Table 3 gives the static indices for detergent compositions of this example, which are charged to the washing machine at the rate of 100 grams per load (about 0.155%, on the basis of the wash water). The additional weights of neodecano-
20. amide employed (0 g, 3 g, 5 g, and 10 g) are given in Table 3. The N-alk(en)yl neoalkanoamides will be sprayed onto the detergent beads as liquids at room temperature or at elevated temperatures, but can be applied in solvents and may be mixed with the
25. detergent as a powder, alone or with a carrier. In cases wherein multiple determinations were made, averages are given.

Table 3

	Alk(en)yl of N-alkenyl <u>Neodecanamide</u>	0 g/wash (control)	3 g/wash	5 g/wash	10 g/wash
5.	Methyl	38	-	-	43
	Ethyl	33	-	37	40
	t-butyl	-	-	-	41
	Octyl	41	-	26	7
10.	Coco alkyl (prime neodecanoic acid reactant)	42	26	14	5
	Coco alkyl (tech- nical neodecanoic acid reactant)	45	19	11	-
15.	Myristyl	31	-	11	-
	Palmityl	43	-	-	26
	Tallow alkyl	41	-	21	16
	Oleyl	-	32	17	10
20.	Stearyl	-	-	-	28
	Hydrogenated tallow alkyl	46	-	-	32
25.	Alk(en)yl of N- alk(en)yl neo- <u>pentanoamide</u>				
	Tallow alkyl	46	-	-	20

30.

When skilled evaluators of fabric properties examine the test fabrics washed with control and experimental detergent compositions for static cling they note no improvement for the N-methyl neodecano-
 5. amide, N-ethyl neodecanoamide and N-t-butyl neodecanoamide, but significant improvements are found for all the other neodecanoamides reported in Table 3, at the various concentrations indicated.

Reflectometer readings of the soil removal index
 10. swatches showed no adverse effects on soil removal by the tested antistats. Similarly, the blue cloth was not adversely lightened in colour, stained, or otherwise undesirably changed in appearance, compared to a control.

15. When the other neoalkanoamides described in this specification are tested in similar manner it will be found that they too impart desirable antistatic characteristics to washed laundry. Similarly, when other detergent compositions are utilised, such as

20. non-phosphate detergent compositions containing zeolite builder, and nonionic detergent compositions, with or without phosphate builder and with or without zeolite builder, similar results are also obtainable. In addition to the particulate detergent compositions,

25. liquid detergent compositions may also be employed, in which the neodecanoamide may be dissolved and/or dispersed, or with which it may be added to the wash water in a washing machine. One such formula

includes 16% of Neodol 25-7 (condensation product of
 30. one mole of higher fatty alcohol of 12 to 15 carbon

atoms with 7 moles of ethylene oxide), 5.5% of denatured alcohol (3A), 3.1% of sodium linear dodecylbenzene sulphonate, 0.2% of fluorescent brightener, 3% of sodium formate, 1% (active ingredient basis) of soil release promoting agent (Alkaril QCJ), 0.8% of enzymes, 0.01% of blue dye, 0.4% of perfume, 10% of N-coco alkyl neodecanoamide and 60% of water. It may also be desirable to incorporate about 5 to 10% of a hydrotrope, such as sodium benzene sulphonate, in the formula to stabilise it against settling out of the antistat.

EXAMPLE 6

Benonite powder, of a fineness of about No. 200 (U.S. Sieve Series, (which has openings 74 microns across), is agglomerated in known manner by tumbling it in an inclined drum while spraying onto falling curtains of the powder a relatively dilute aqueous sodium silicate solution (about 2%) until agglomerate beads of about the desired size result. Such beads are dried to acceptable moisture content, which may be about 11%. Thereafter the beads are screened to desired size range, such as Nos. 10 to 60, U.S. Sieve Series (which have openings 200 to 250 microns across). N-tallow alkyl neodecanoamide, which is an oily liquid at room temperature, is sprayed onto the surfaces of the bentonite agglomerate beads while such beads are being tumbled in an inclined drum, and the porous bentonite beads absorb the neodecanoamide so that the resulting particulate product remains free flowing. The proportion of neodecanoamide in

the product may be varied as desired but is desirably about 20%.

When one part of the N-tallow alkyl neodecano-
amide-bentonite agglomerate product is mixed with
5 three parts of the detergent composition of Example 5
(that without neoalkanoamide) the resulting composi-
tion contains 20% of bentonite and 5% of the neo-
alkanoamide. Laundry washed with such composition,
at a concentration in the wash water of about 0.2%,
10 is noticeably soft (especially important for cotton
items) and does not exhibit objectionable static
cling (important for synthetic items). The laundry
softening capability of the composition is greater
than would have been attributed to the bentonite
15 content thereof and it is considered that the
N-tallow alkyl neodecanoamide, in the presence of
bentonite, either increases the bentonite softening
activity or possesses independent softening charac-
teristics. Similar bentonite-neoalkanoamide products
20 made with others of the described neoalkanoamides do
not exhibit the softening increasing effect with the
N-tallow alkyl neodecanoamide, but they are useful
for their antistatic properties, in conjunction with
the fabric softening activity of the bentonite.

25 Instead of spraying liquid state neoalkanoamide
onto the bentonite agglomerate it may be sprayed onto
the finely divided powder before agglomeration, and
may assist in agglomerating the bentonite. Also, for
the neoalkanoamides that are in solid state at room
30 temperature mixtures of such in powdered form with
benonite powder may be made. Alternatively, such
materials may be liquefied by means of heat or

solvents, and may be applied to bentonite agglomerates or powders in such state.

Instead of employing the bentonite-neoalkanoamide composition as an additive to a detergent composition to make it softening and antistatic, that composition may be used to treat already washed material, as in rinse water. The neoalkanoamide, such as N-coco alkyl neodecanoamide, N-myristyl neodecanoamide, N-oleyl neodecanoamide or N-octyl neodecanoamide, may be added to either the wash water, rinse water or other aqueous treating medium in a sufficient concentration, normally in the range of 0.005 to 0.1%, to impart antistatic properties to fibrous materials. In such instances it will be normal to machine dry the fibrous material or laundry, as in a tumbling automatic laundry dryer. It is known that is the act of drying the laundry while moving it which promotes the development of static charges thereon, which development is inhibited by treatment with a neoalkanoamide of the present invention.

Although satisfactory antistatic action is obtained when the present neoalkanoamides are employed in or with built laundry detergent compositions, and fabric softening is obtained from N-tallow alkyl neoalkanoamide when it is employed with bentonite in or with detergent compositions, better antistatic action is noted and better softening effects for the neoalkanoamides are obtained with tallow alkyl neodecanoamide (with bentonite) when such are employed in rinsing steps, rather than in washing operations.

Wash cycle additive products of various types may be made, including particulate, paste, gel, liquid and solid tablet products, with the particulate material often preferably including

5 inorganic builder salt, sodium sulphate, agglomerated bentonite and perfume, as well as neoalkanoamide, which preferably is coco alkyl neodecanoamide or tallow alkyl neodecanoamide. One useful formula for such a particulate products includes: 66.8% of spray

10 dried base beads made by spray drying, to a moisture content of about 8%, a crutcher mix containing 34.2 parts of water, 1.4 parts of fluorescent brightener, 1 part of magnesium sulphate monohydrate, 0.4 part of sodium polyacrylate, 32 parts of zeolite 4A, 2.5

15 parts of white montmorillonite (or bentonite), 17 parts of sodium bicarbonate (during which spray drying half of the sodium bicarbonate is converted to sodium carbonate), and 11.5 parts of soda ash; 21

20 parts of N-coco alkyl neodecanoamide or N-tallow alkyl neodecanoamide; 0.2 part of perfume; and 12 parts of agglomerated bentonite (10-60 mesh) (which have openings 200 to 250 microns across).

Instead of the base materials that have been recited for application of the neoalkanoamide, other

25 carriers may be employed, such as Microcel C (synthetic calcium silicate), sodium sulphate, soda ash or borax. Also, the neoalkanoamide may be dissolved or dispersed in an aqueous, alcoholic or other suitable solvent medium and applied alone or

30 with adjuvant(s). In some instances it may be

desirable to spray a normally liquid state neoalkanoamide onto surfaces to be made antistatic.

A liquid preparation for addition to the rinse water to render washed laundry antistatic may include

5. about 91 parts of distilled water, about 1 part of perfume, about 0.3 part of nonionic detergent (preferably Neodol 25-7), about 2 parts of isopropanol and about 5.7 parts of N-coco alkyl neodecanoamide, N-tallow alkyl neodecanoamide or
10. other suitable neoalkanoamide within the present invention.

EXAMPLE 7

A non-woven rayon sheet is impregnated with about 1.5 times its weight of coconut oil fatty acids

15. diglyceride, about 1/2 its weight of N-coco alkyl neodecanoamide (or N-tallow alkyl neodecanoamide or other suitable neoalkanoamide) and a desirable proportion (0.5%) of perfume. This product is useful as an antistatic sheet to be added to a laundry
20. dryer, with the charge of sheeting to the dryer being such that about 10 grams of the neoalkanoamide will be present therein for every 5 to 10 pounds (1 to 22 kg) of laundry (dry basis).

- Alternatively, the neoalkanoamide may be applied
25. to the tumbling drying laundry by other mechanisms, including dispensing from a sponge or spraying or dripping liquid state neoalkanoamide onto the tumbling laundry in the automatic dryer.

- In the above description and examples it is to
30. be understood that mixtures of the neoalkanoamides of

the present invention may be employed, usually with the adjustment of the composition being such as to obtain most satisfactory antistatic action. Also, application conditions and proportions may be
5 adjusted to obtain the desired results in the particular circumstances.

The invention has been described with respect to various embodiments and illustrations thereof, but is not to be limited to these because it is evident that
10 one of skill in the art will be able to utilise substitutes and equivalents without departing from the invention claimed.

15

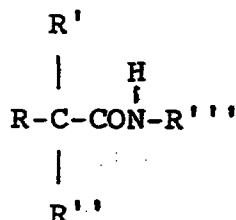
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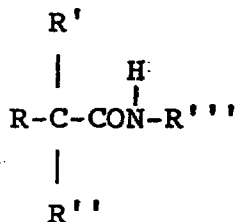
CLAIMS

1. Antistatic compositions containing N-higher alk(en)yl neoalkanoamides having the formula



in which R, R' and R'' each represent alkyl groups which may be the same or different and in which the sum of the carbon atoms in the groups R, R' and R'' is 3 to 14 and R''' represents an alkyl or alkenyl group having 8 to 20 carbon atoms.

2. N-higher alk(en)yl neoalkanoamides having the formula



in which R, R' and R'' each represent alkyl groups which may be the same or different and in which the sum of the carbon atoms in the groups R, R' and R'' is 3 to 14 and R''' represents an alkyl or alkenyl group having 8 to 20 carbon atoms, the R''' groups being linear.



3. N-Higher alk(en)yl neoalkanoamides as claimed in Claim 2 in which the higher alk(en)yls, R''', are alkyl groups of an average of 12 to 18 carbon atoms.

4. N-higher alk(en)yl neoalkanoamides as claimed in Claim 2 or Claim 3 in which the R''' groups are coco alkyl.

5. N-higher alk(en)yl neoalkanoamides as claimed in Claim 2 or Claim 3 in which the R''' groups are tallow alkyl.

6. N-higher alk(en)yl neoalkanoamides according to Claim 2 or Claim 3 in which the R''' groups are hydrogenated tallow alkyl.

7. N-higher alk(en)yl neoalkanoamides as claimed in any one of Claims 2 to 6 which are neodecanoamides.

8. N-higher alk(en)yl neoalkanoamides as claimed in any one of Claims 2 to 7 in which the sum of the carbon atoms in the groups R, R' and R'' is 5 to 12.

9. N-myristyl neodecanoamide.

10. N-palmityl neodecanoamide.

11. N-oleyl neodecanoamide.

12. N-tallow alkyl neopentanoamide.

13. N-higher alk(en)yl neoalkanoamides as claimed in Claim 2 substantially as specifically described



herein with reference to the examples and the accompanying Figures.

14. A process for the production of N-higher alk(en)yl neoalkanoamide as a light in colour oil which comprises reacting a higher alk(en)yl amine with neoalkanoic acid of 5 to 16 carbon atoms at an elevated temperature under an inert gas or nitrogen atmosphere or under vacuum, and separating the product resulting from impurities, by-products and unreacted amine and neoalkanoic acid.

15. A process as claimed in Claim 14 substantially as specifically described herein with reference to the Examples.

16. N-higher alk(en)yl neoalkanoamides as claimed in Claim 2 whenever made by a process as claimed in Claim 14 or Claim 15.

17. A detergent composition comprising a deterative proportion of a synthetic organic detergent and a proportion, which is antistatic characteristic imparting to laundry during washing, of an N-higher alk(en)yl neoalkanoamide as claimed in any one of Claims 2 to 13 or 16 or an antistatic composition as claimed in Claim 1.

18. A detergent composition as claimed in Claim 17 which is in particulate form and which comprises about 5 to 35% of synthetic organic detergent of sulphate and/or sulphonate type, about 10 to 85% of builder for such synthetic organic detergent, about 1



to 20% of the N-higher alk(en)yl neoalkanoamide, about 2 to 20% of moisture, and with the balance, if any, of filler(s) and/or adjuvant(s), with the particles of the detergent composition being in the Nos. 10 to 140 range, U.S. Sieve Series (which have openings 2000 to 105 microns across).

19. A detergent composition as claimed in Claim 17 or Claim 18 in which the N-higher alk(en)yl neoalkanoamide is N-higher alkyl neodecanoamide.

20. A detergent composition as claimed in Claim 17, 18 or 19 in which the higher alkyl of the N-higher alkyl of the N-higher alkyl neodecanoamide is of an average number of carbon atoms in the range of 12 to 18.

21. A detergent composition as claimed in Claim 17, 18, 19 or 20 in which the proportion of N-higher alk(en)yl neoalkanoamide in the detergent composition is from 7 to 10%.

22. A detergent composition as claimed in any one of Claims 17 to 21 in which the builder is selected from the group consisting of polyphosphates, carbonates, bicarbonates, sesquicarbonates, silicates, sesquisilicates, citrates, nitrilotriacetates, polyacetal carboxylates, zeolites, and mixtures thereof.

23. A detergent composition as claimed in any one of Claims 17 to 22 in which the synthetic organic detergent is selected from the group consisting of



linear higher alkylbenzene sulphonates, branched higher alkylbenzene sulphonates, higher fatty alcohol sulphates, olefin sulphonates, paraffin sulphonates, monoglyceride sulphates, higher fatty alcohol ethoxylate sulphates, higher fatty acid sulphoesters of isethionic acid, higher fatty acyl sarcosides, and acyl- and sulpho-amides of N-methyltaurine.

24. A detergent composition as claimed in any one of Claims 17 to 23 in which the N-higher alkyl neodecanoamide is N-tallow alkyl neodecanoamide, which composition is of improved fabric softening properties compared to such properties of a composition otherwise the same but containing only bentonite and no N-higher alk(en)yl neodecanoamide, the N-tallow alkyl neodecanoamide being present in the detergent composition dispersed in a greater proportion of bentonite, the mixture of bentonite and N-tallow alkyl neodecanoamide being mixed with other components of the detergent composition, the said other components being in spray dried bead form.

25. A blend of bentonite and N-tallow alkyl neodecanoamide.

26. A detergent composition as claimed in any one of Claims 17 to 24 containing a blend of bentonite and N-tallow alkyl neodecanoamide.

27. A process for washing laundry and simultaneously lowering its capacity to generate and/or hold an electrostatic charge, which comprises washing the laundry in an aqueous medium containing a deterative



* C L M S *

proportion of a detergent composition and an antistatic proportion of an N-higher alk(en)yl neoalkanoamide as claimed in any one of Claims 2 to 13 or 16 or an antistatic composition as claimed in Claim 1 rinsing the laundry and drying it.

28. A process as claimed in Claim 27 in which the deterative proportion of the detergent composition in the aqueous medium is from 0.05 to 0.5%, the antistat is N-higher alkyl neodecanoamide, and the antistatic proportion of N-higher alkyl neodecanoamide is in the range of 0.005 to 0.1%.

29. A process for treating laundry to lower its capacity to generate and/or hold an electrostatic charge which comprises washing the laundry in a wash water containing a deterative proportion of a detergent composition, and rinsing the laundry with rinse water containing an antistatic proportion of an N-higher alk(en)yl neoalkanoamide antistatic agent as claimed in any one of Claims 2 to 13 or 16, or an antistatic composition as claimed in Claim 1.

30. A process as claimed in Claim 29 in which the antistatic proportion of N-higher alkyl neodecanoamide in the rinse water is in the range of 0.005 to 0.1%.

31. A process as claimed in Claim 27 or Claim 29 substantially as specifically described herein with reference to the accompanying examples.

32. A wash cycle additive composition or a rinse or other laundry treating composition comprising an N-higher alk(en)yl neoalkanoamide as claimed in any one of Claims 2 to 13 or 16, or an antistatic composition



as claimed in Claim 1.

33. A composition as claimed in Claim 32 for use in a laundry drier to deposit the said N-higher alk(en)yl neoalkanoamide on the laundry.

34. A composition as claimed in Claim 33 in which the N-higher alk(en)yl neoalkanoamide is deposited on a substrate in an amount of 10 to 100% by weight of the substrate.

35. A rinse composition as claimed in Claim 32 in which the N-higher alk(en)yl neoalkanoamide is dissolved in a solvent or dispersed in an aqueous medium.



36. A rinse composition as claimed in Claim 35 containing 5 to 12% of the N-higher alk(en)yl neoalkanoamide, 30 to 90% of the solvent or aqueous medium and optionally 0.1 to 5% of hydrotone or surface active material.

37. A composition as claimed in any one of Claims 32 to 36 which also contains a quaternary ammonium halide, the proportion of quaternary compound to neoalkanoamide being in the range 1:0.5 to 1:10.

38. A composition as claimed in Claim 32 comprising a solid carrier onto which the N-higher alk(en)yl neoalkanoamide is absorbed.

39. A composition as claimed in any one of Claims 32 to 38 also containing bentonite.

40. A composition as claimed in Claim 38 in which the carrier is porous bentonite beads into which N-higher alk(en)yl neoalkanoamide is absorbed.

41. A composition as claimed in Claim 38 in which the carrier is synthetic calcium silicate, sodium sulphate, borax or soda ash.

42. A rinse composition as claimed in Claim 32 comprising 91 parts water, 1 part perfume, 0.3 parts nonionic detergent, 2 parts isopropanol and 5.7 parts of N-higher alk(en)yl neoalkanoamide.

43. A laundry drier composition as claimed in



Claim 32 comprising a non woven rayon sheet carrying 1.5 times its weight of coconut oil fatty acids diglyceride, and 0.5 times the sheets weight of N-higher alk(en)yl neoalkanoamide.

5

44. A wash cycle additive composition as claimed in any one of Claims 32 to 43 in particulate, paste, gel, liquid or tablet form.

10

45. A wash cycle additive as claimed in Claim 44 in particulate form including one or more of inorganic builder salt, sodium sulphate and agglomerated bentonite.

15

46. A wash cycle additive as claimed in Claim 44 or Claim 45 in which the composition contains spray dried beads.

47. A wash cycle additive as claimed in Claim 46 comprising 66.8% of spray dried base beads made by spray drying to a moisture content of about 8%, a crutcher mix containing 34.2 parts of water, 1.4 parts of fluorescent brightener, 1 part of magnesium sulphate monohydrate, 0.4 part of sodium polyacrylate, 32 parts of zeolite 4A, 2.5 parts of white montmorillonite (or bentonite), 17 parts of sodium bicarbonate (during which spray drying half of the sodium bicarbonate is converted to sodium carbonate), and 11.5 parts of soda ash; 21 parts of N-coco alkyl neodecanoamide or N-tallow alkyl neodecanoamide; 0.2 part of perfume; and 12 parts of agglomerated bentonite (10-60 mesh) (which have openings 200 to 250 microns across).

