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|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |           |                                                                                                                                                                                                          |
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| <b>(54) Title:</b> OPTICAL CLARITY STABILIZING FATTY ALKYL DIETHANOLAMINES<br><br><b>(57) Abstract</b><br><br>The tendency to opacify of a polyol composition comprising one or more zinc fatty acid salts such as zinc stearate, added to form a mold release agent when the polyol is converted to a molded polyurethane article, is retarded by the presence of a small but effective amount of one or more fatty alkyl diethoxylate (e.g. diethanolamine) compounds.                                                                                                                                                                          |           |                                                                                                                                                                                                          |

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# 1 OPTICAL CLARITY STABILIZING FATTY ALKYL DIETHANOLAMINES

The present invention relates to the  
5 stabilization of certain liquid organic compositions  
of matter against opacification, that is, against the  
tendency of such liquid compositions over time to lose  
optical clarity and/or to become progressively less  
translucent.

10 More specifically, the present invention  
relates to the treatment of liquid compositions of  
matter comprising polyols, and especially containing  
as well amine-terminated polyols, already containing  
as an additive one or more zinc salts of fatty acid,  
15 to retard the tendency of such compositions to opacify  
with the passage of time.

Polyols are used, among other things, in the  
synthesis of polyurethane polymers. In general,  
polyols, comprising one or a mixture of organic  
20 compounds substituted with two or more hydroxyl groups  
are reacted under appropriate conditions with  
isocyanates containing two or more isocyanate  
functionalities, with the reaction between the  
hydroxyl groups and the isocyanate functionalities  
25 leading to the formation as desired of a polymer  
network connected by urethane linkages. The resulting  
polymeric products are conventionally referred to as  
polyurethanes.

Solid articles, including both rigid objects  
30 as well as flexible and foamed articles, are often  
manufactured in molds wherein the polyol and

1 isocyanate reactants, together with catalysts and  
other additives as desired, are combined in the mold  
whereupon the polyurethane that forms in that mold  
takes the shape of the desired article. Experience  
5 has shown, however, that the polyurethane material can  
tend to adhere to the inner surfaces of the mold with  
a tenacity such that removing the article from the  
mold can require application of considerable force, or  
can require the use of specialized tools and/or the  
10 disassembly of the mold itself. Accordingly, it is  
known to incorporate amongst the components that  
together form the polyurethane an effective amount of  
one or more "internal" mold release agents capable of  
reducing the adhesion between the formed polyurethane  
15 surface and the contiguous surface of the mold so that  
separation and removal of the polyurethane article  
from the mold are facilitated without undue  
complication and without damage to the surface of the  
polyurethane article. Among the compounds which have  
20 been found in practice to be effective mold release  
agents are zinc salts of fatty acids, typically  
saturated or unsaturated fatty acids containing 8 to  
24 carbon atoms, one notable example of which is zinc  
stearate.

25 For instance, it is typical to incorporate a  
zinc fatty acid salt, such as zinc stearate, into the  
polyol component precursor of the reaction which forms  
the polyurethane. Unfortunately, it has been found  
that the presence of zinc salts of fatty acids, such  
30 as zinc stearate, even at the relatively low amounts  
on the order of 0.5 to 10 wt.% based on the polyol in

1 which such mold release agents are present, appears to  
give to the polyol composition a tendency to opacify  
with the passage of time, over even one day or a few  
days. That is, the composition of matter comprising  
5 polyol and zinc fatty acid salt gradually loses  
translucency. This tendency is undesirable for  
several reasons. One reason is the aesthetic, in that  
a relatively hazy product will appear relatively  
unattractive to the customer and will suggest that the  
10 polyol component itself may also have suffered some  
degradation or loss of desired reactivity. In  
addition, the opacification may be due to some  
undefined interaction with the polyol itself, such  
that a relatively hazier product may well have a lower  
15 content of active polyol capable of entering into the  
desired reaction forming the polyurethane than is the  
case with a clear polyol composition. In addition,  
the haze is evidently due to the presence of a  
separate, denser or solid phase, which even if it is  
20 in the form of an ultrafine and dispersed solid may  
nonetheless interfere with processing and reactor  
equipment, lines, and valves, and may be expected to  
detract from the appearance and the property of the  
polyurethane product which is subsequently formed.

25           There thus remains a need for a technique  
for alleviating the tendency of polyol compositions  
containing zinc fatty acid salts to opacify over time.  
The present invention has been found to fulfill this  
need and to exhibit the other desired properties  
30 described herein.

1           The present invention is directed to a  
process for retarding the tendency to opacify of a  
liquid composition of matter comprising polyol as the  
major component and also comprising one or more zinc  
5 salts of fatty acids containing 8 to 24 carbon atoms,  
comprising forming a composition of matter comprising  
said polyol as the major component, said one or more  
zinc salts of fatty acids containing 8 to 24 carbon  
atoms, and one or more fatty alkyl diethoxylates in a  
10 small amount effective to retard said tendency to  
opacify, wherein said fatty alkyl diethoxylates have  
the formula  $R-N((-CH_2CH_2O)_nH)_2$  in which R is alkyl or  
alkenyl containing 12 to 22 carbon atoms, and n is 1  
to 5.

15           The present invention is further directed to  
a composition of matter comprising one or more zinc  
salts of fatty acid containing 8 to 24 atoms, and one  
or more fatty alkyl diethoxylates of the formula  $R-N((-CH_2CH_2O)_nH)_2$  wherein R is alkyl or alkenyl containing  
20 12 to 22 carbon atoms, and n is 1 to 5; in which said  
one or more fatty alkyl diethoxylates comprise about 1  
to about 10% by weight of the amount of said one or  
more zinc salts of fatty acid.

          Still further, the present invention is  
25 directed to a composition of matter comprising a major  
amount of polyol and further comprising a composition  
as described in the preceding paragraph in an amount  
thereof such that about 0.1 to about 10 wt.% of said  
composition of matter comprises said one or more zinc  
30 salts of fatty acids containing 8 to 24 carbon atoms.

1 Polyol compositions with which the present  
invention is useful extend to any liquid composition  
containing any one or more polyols, wherein the  
presence of zinc stearate or other zinc fatty acid  
5 salt leads to gradual opacification over time, i.e.  
over the passage of up to thirty days. Such polyols,  
including blends of polyols, thus include polyols used  
in the synthesis of polyurethane.

These compounds may be divided into two  
10 groups, high molecular weight compounds having a  
molecular weight of 400 to about 10,000 and low  
molecular weight compounds, i.e. chain extenders,  
having a molecular weight of 62 to 399. Examples of  
suitable high molecular weight compounds include the  
15 polyesters, polyethers, polythioethers, polyacetals  
and polycarbonates containing at least 2, preferably 2  
to 8 and most preferably 2 to 4 isocyanate-reactive  
groups of the type known for the production of  
polyurethanes.

20 The high molecular weight polyethers  
suitable for use in accordance with the invention are  
known and may be obtained, for example, by  
polymerizing epoxides such as ethylene oxide,  
propylene oxide, butylene oxide, tetrahydrofuran,  
25 styrene oxide or epichlorohydrin in the presence of  
BF<sub>3</sub> or by chemically adding these epoxides, preferably  
ethylene oxide and propylene oxide, in admixture or  
successively to components containing reactive  
hydrogen atoms such as water, alcohols or amines.  
30 Examples of alcohols and amines include the low  
molecular weight chain extenders set forth

1 hereinafter, 4,4'-dihydroxy diphenyl propane, sucrose,  
aniline, ammonia, ethanolamine and ethylene diamine.

It is preferred to use polyethers which contain  
substantial amounts of primary hydroxyl groups in  
5 terminal positions (up to 90% by weight, based on all  
of the terminal hydroxyl groups present in the  
polyether). Polyethers modified by vinyl polymers, of  
the type formed, for example, by polymerizing styrene  
or acrylonitrile in the presence of polyether (U.S.  
10 Patent Nos. 3,383,351; 3,304,273; 3,523,093; and  
3,110,695; and German Patent 1,152,536), are also  
suitable, as are polybutadienes containing OH groups.

In addition, polyester polyols which contain  
high molecular weight polyadducts or polycondensates  
15 in finely dispersed form or in solution may be used.  
Such modified polyether polyols are obtained when  
polyaddition reactions (e.g. reactions between  
polyisocyanates and amino functional compounds) or  
polycondensation (e.g., between formaldehyde and  
20 phenols and/or amines) are directly carried out in  
situ in the polyether polyols.

Suitable examples of high molecular weight  
polyesters include the reaction products of  
polyhydric, preferably dihydric alcohols (optionally  
25 in the presence of trihydric alcohols), with  
polyvalent, preferably divalent, carboxylic acids.  
Instead of using the free carboxylic acids, it is also  
possible to use the corresponding polycarboxylic acid  
anhydrides or corresponding polycarboxylic acid esters  
30 of lower alcohols or mixtures thereof for producing  
the polyesters. The polycarboxylic acids may be

1 aliphatic, cycloaliphatic, aromatic, and/or  
heterocyclic and may be unsaturated or substituted,  
for example, by halogen atoms. The polycarboxylic  
acids and polyols used to prepare the polyesters are  
5 known and described for example in U.S. Patent Nos.  
4,098,731 and 3,726,952. Suitable polythioethers,  
polyacetate, polycarbonates and other polyhydroxyl  
compounds are also disclosed in the above identified  
U.S. patents. Finally, representatives of the many  
10 and varied compounds which may be used in accordance  
with the invention may be found for example in High  
Polymers, Volume XVI, "Polyurethanes, Chemistry and  
Technology", by Saunders-Frisch, Interscience  
Publishers, New York, London, Vol. I, 1962, pages 32-  
15 42 and 44-54, and Volume II, 1964, pages 5-6 and 198-  
199; and in Kunststoff-Handbuch, Vol. VII, Vieweg-  
Hoochtlen, Carl Hahset Verlag, Munich, 1966, pages 45-  
71.

Suitable aminopolyethers which may be used  
20 in accordance with the present invention as high  
molecular weight compounds ( the molecular weight is  
always the average molecular weight which may be  
calculated from the functionality and the content of  
isocyanate-reactive groups) are those wherein at least  
25 about 30 and preferably about 60 to 100 equivalent %  
of the isocyanate-reactive groups are primary and/or  
secondary (preferably primary), aromatically or  
aliphatically (preferably aromatically) bound amino  
groups and the remainder are primary and/or secondary  
30 aliphatically bound hydroxyl groups.

1           In these compounds, the terminal residues  
carrying the amino groups may also be attached to the  
polyether chain by urethane or ester groups. These  
"aminopolyethers" are prepared by methods known per  
5 se. For example, polyhydroxypolyethers such as  
polypropylene glycol ethers may be aminated by  
reaction with ammonia in the presence of Raney nickel  
and hydrogen (BE-PS 634,741). U.S. Patent No.  
3,654,370 describes the production of polyoxyalkylene  
10 polyamines by reaction of the corresponding polyol  
with ammonia and hydrogen in the presence of a nickel,  
copper, chromium catalyst. DE-PS 1,193,671 describes  
the production of polyethers containing terminal amino  
groups by hydrogenation of cyanoethylated  
15 polyoxypropylene ethers. Other methods for the  
production of polyoxyalkylene (polyether) amines are  
described in U.S. Patent Nos. 3,155,728, and 3,236,895  
and FR-PS 1,551,605. The production of polyethers  
containing terminal secondary amino groups is  
20 described, for example, in FR-PS 1,466,708.

Polyhydroxypolyethers of relatively high  
molecular weight may be converted into the  
corresponding anthranilic acid esters by reaction with  
isatoic acid anhydride, as described, for example, in  
25 DE-OS Nos. 2,019,432 and 2,619,840 and in U.S. Patent  
Nos. 3,808,250, 3,975,428 and 4,016,143. Polyethers  
containing terminal aromatic amino groups are formed  
in this way.

According to DE-OS 2,546,536 and U.S. Patent  
30 No. 3,865,791, relatively high molecular weight  
compounds containing terminal amino groups are

1 obtained by reaction of NCO prepolymers based on  
polyhydroxypolyethers with enamines, aldimines or  
ketimines containing hydroxyl groups and subsequent  
hydrolysis.

5 Amino polyesters obtained by hydrolysis of  
compounds containing terminal isocyanate groups, for  
example in accordance with DE-OS 2,948,419 or U.S.  
Patent No. 4,515,923, can also be present. In this  
process, polyethers most preferably containing 2 to 4  
10 hydroxyl groups are reacted with polyisocyanates to  
form NCO prepolymers and, in a second step, the  
isocyanate groups are converted by hydrolysis into  
amino groups.

The "aminopolyethers" used in accordance  
15 with the invention are often mixtures of the compounds  
mentioned by way of example and (on a statistical  
average) most preferably contain 2 to 4 terminal  
isocyanate-reactive groups. In the process according  
to the invention, the "aminopolyethers" may be used in  
20 admixture with polyhydroxypolyethers free from amino  
groups.

In accordance with the present invention,  
the high molecular weight compounds are used in  
admixture with up to about 95% by weight, preferably  
25 up to about 50% by weight, more preferably about 8 to  
30% by weight and most preferably about 12 to 26% by  
weight, based on the total quantity of the high  
molecular weight compounds, of the low molecular  
weight chain extenders. Examples of suitable hydroxyl  
30 group-containing chain extenders include ethylene  
glycol, 1,2- and 1,3-propane diol, 1,3- and 1,4- and

1 2,3-butane diol, 1,6-hexane diol, 1,10-decane diol,  
diethylene glycol, trimethylene glycol, tetraethylene  
glycol, trimethylene glycol, tetraethylene glycol,  
dipropylene glycol, tripropylene glycol, glycerol and  
5 trimethylol propane.

While not intending to be bound by any particular theory of the operation of the present invention, the tendency of such polyols to opacify over time when a zinc fatty acid salt such as zinc  
10 stearate is present in the polyol, is consistent with the theory that the zinc salt is relatively insoluble in the polyol (whether the polyol is but one structure or comprises a blend of two or more distinct compounds). Thus, the polyols to which the discovery  
15 of the present invention is applicable extend to any polyol compositions in which the amount of zinc fatty acid salt to be incorporated for its intended property as a mold release agent approaches or exceeds the solubility limit of the zinc fatty acid salt in the  
20 polyol composition. For practical purposes, this would mean any polyol compositions wherein the amount of the zinc fatty acid salt component is greater than 50% of its solubility limit in the polyol composition.

In these compositions of matter, the one or  
25 more polyol compounds can comprise the entire composition (other than the zinc fatty acid salt and the fatty amine derivative added in accordance with the present invention,) but it will be recognized that other components and diluents may be present as  
30 desired. In any event, the polyol component (which, as used herein, refers to the entirety of the one or

1 more polyol compounds present) will constitute the  
major amount of the combination of matter, i.e.  
generally at least about 80 wt.%, preferably at least  
90 wt.% and more preferably at least 95 wt.% of the  
5 composition to which the present invention is applied.

The zinc fatty acid salts customarily added  
to the polyol composition intended to function as a  
mold release agent for the subsequently formed  
polyurethane, comprise zinc salts of one or more fatty  
10 acids. As used herein, "fatty acids" refers to  
saturated and unsaturated, straight and branched  
carboxylic acids containing 8 to 24 carbon atoms. The  
most preferred of such zinc salts is zinc stearate.  
Other preferred zinc salts includes zinc palmitate,  
15 zinc laurate, and zinc octoate.

The amount of the zinc salt component  
depends generally on the characteristics of the  
particular polyurethane to be manufactured. Amounts  
of 0.1 wt.% to about 10 wt.% of zinc fatty acid salts,  
20 expressed as a weight percentage of the polyol  
composition, are typically employed. Preferred  
amounts of the zinc fatty acid salts are generally  
about 0.5 to 5 wt.%, and more preferably about 1 to  
about 4 wt.%.

25 The present invention uses to advantage a  
fatty amine diethoxylate component, by which is meant  
one or more compounds of the general formula  
 $R-N((-CH_2CH_2O)_nH)_2$  wherein R denotes a straight-chain or  
branched-chain, saturated or unsaturated, alkyl or  
30 alkenyl chain containing 12 to 22 carbon atoms.  
Preferably, R denotes a stearyl chain. In the

1   aforementioned formula each n is 1 to 5, and  
preferably 1. Thus, a most preferred diethoxylate is  
stearyl diethanol amine. Such compounds are generally  
commercially available, or can readily be synthesized  
5   from a corresponding alkyl amine by reaction with an  
appropriate stoichiometric amount of ethylene oxide.

The particular sequence of combination of  
fatty amine diethoxylate and zinc fatty acid salt to  
the polyol composition is not critical and addition  
10   may be carried out in any order convenient to the  
operator. A discrete product comprising a combination  
of one or more zinc salts of fatty acid and one or  
more fatty amine diethoxylate can be formed, by  
appropriate heating and stirring to a uniform  
15   distribution of the components. This product can then  
be thoroughly stirred into the polyol, with heating of  
the composition of matter as necessary to thoroughly  
liquify the composition, all so as to insure complete  
and uniform distribution of the zinc fatty acid salt  
20   and the fatty amine diethoxylate throughout the polyol  
composition. Alternatively, the zinc fatty acid salt  
and the fatty amine diethoxylate can each be stirred  
into the polyol composition under conditions selected  
to afford a thorough and uniform distribution of each  
25   component throughout the polyol composition. Heating  
may be necessary to improve the liquidity and aid the  
desired thorough distribution of components.

The amount of fatty amine diethoxylate  
should comprise a sufficient quantity so as to retard  
30   the aforementioned opacification of the polyol  
composition of matter containing the zinc fatty acid

1 salt which would otherwise be observed. Generally,  
amounts of fatty amine diethoxylate on the order of  
about 1% to about 10% by weight of the amount of zinc  
fatty acid salt present will be useful. More  
5 typically, the ratio by weight of fatty amine  
diethoxylate to zinc fatty acid salt should preferably  
lie in the range of about 1:10 to 1:25.

Thus, it can be seen that the zinc fatty  
acid salt and the fatty amine diethoxylate comprise  
10 minor amounts of the polyol composition, each  
generally comprising from about 0.1 wt.% to about 5  
wt.% thereof with the fatty amine diethoxylate itself  
comprising generally up to about 1 wt.% of the overall  
polyol composition.

15 The present invention is further described  
in the following Example, which is provided for  
purposes of illustration and is not intended to limit  
the scope of that which is regarded as the invention.

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EXAMPLE

The tests described in this example evaluated the effect of adding fatty amine diethoxylate to samples of a polyol composition to which zinc fatty acid salt had also been added. The fatty amine diethoxylate was added to only certain samples, to compare the opacification with and without the addition of the fatty amine diethoxylate.

10 The procedure was as follows: a mold release concentrate was prepared which contained about 28.6 wt.% of zinc fatty acid salt, whose identity in each run is set forth in Table A below. Then, for each different concentrate, 80 grams of a polyol  
15 composition to be subsequently reacted in the synthesis of polyurethane was placed in an 8 ounce jar, after which 4 grams of the given mold release concentrate was added to the same jar and the contents were mixed together thoroughly until the mixture was  
20 completely clear and free of lines of convection, indicating uniformity of distribution of the components. Then, 20 grams of this mixture was placed into each of 3 sample bottles which were then capped securely and placed in an air-circulating oven preset  
25 to a constant temperature of 94°F. The samples were then checked daily, and the day upon which a sample was first seen to be hazy (indicating excessive opacity) was noted.

The results of each test are set forth below  
30 in Table A which shows the run number, the identity of the particular zinc fatty acid salt employed, and the

1 number of days the sample was able to remain in the oven before it opacified.

TABLE A

| 5  | <u>Test No.</u> | <u>Zinc Salt Composition</u>                            | <u>Days to Haze</u>   |
|----|-----------------|---------------------------------------------------------|-----------------------|
|    | 1               | Zinc Stearate                                           | 5                     |
|    | 2               | Zinc Stearate                                           | 3                     |
| 10 | 3               | Zinc Palmitate                                          | 3                     |
|    | 4               | Zinc Laurate                                            | 20                    |
|    | 5               | 95 wt.% zinc stearate/<br>5 wt.% octoate                | 4                     |
| 15 | 6               | 90 wt.% zinc stearate/<br>10 wt.% zinc laurate          | 11                    |
|    | 7               | 95 wt.% zinc stearate/<br>5 wt.% gluconic acid          | 3                     |
|    | 8               | 95 wt.% zinc stearate/<br>5 wt.% zinc laurate           | 8                     |
| 20 | 9               | 95 wt.% zinc stearate/<br>5 wt.% stearyl diethanolamine | (did not become hazy) |
|    | 10              | Zinc stearate                                           | 4                     |
| 25 | 11              | 95 wt.% zinc stearate/<br>5 wt.% stearyl diethanolamine | (did not become hazy) |

From these results it can clearly be seen that the presence of the fatty amine diethoxylate, in this case stearyl diethanolamine, greatly prolongs the ability of the polyol composition containing zinc

1    stearate to remain clear, resisting opacification.  
Specifically, it can be seen that polyol compositions  
containing zinc stearate without fatty amine  
diethoxylate, such as compositions 1, 2, 3 and 10,  
5    became hazy and opacified within a few days, whereas  
compositions 9 and 11 which also contained the fatty  
amine diethoxylate did not become hazy during the time  
of the testing (that is, not in eight days or more).  
This improvement is clearly useful, and is as well  
10   quite unexpected.

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1 WHAT IS CLAIMED IS

1. A process for retarding the tendency to opacify of a liquid composition of matter comprising polyol as the major component and also comprising one  
5 or more zinc salts of fatty acids containing 8 to 24 carbon atoms, comprising forming a composition of matter comprising said polyol as the major component, said one or more zinc salts of fatty acids containing 8 to 24 carbon atoms, and one or more fatty alkyl  
10 diethoxylates in a small amount effective to retard said tendency to opacify, wherein said fatty alkyl diethoxylates have the formula  $R-N((-CH_2CH_2O)_nH)_2$  in which R is alkyl or alkenyl containing 12 to 22 carbon atoms, and n is 1 to 5.

15 2. The process of claim 1 wherein said one or more fatty alkyl diethoxylates are selected from the group consisting of fatty alkyl diethanolamines wherein the fatty alkyl substituent contains 12 to 22 carbon atoms.

20 3. The process of claim 2 wherein said one or more zinc salts of fatty acids, and said one or more fatty alkyl diethanolamines, each comprise 0.1 to 10 wt.% of said composition of matter.

25 4. The process of claim 3 wherein said composition of matter contains zinc stearate.

5. The process of claim 2 wherein said one or more zinc salts of fatty acid comprise up to about 5 wt.% of said composition of matter.

30 6. The process of claim 5 wherein said one or more fatty alkyl diethanolamines comprise up to about 5 wt.% of said composition of matter.

1                   7. The process of claim 5 wherein said  
fatty alkyl diethanolamine is stearyl diethanolamine.

8. The process of claim 1 wherein said  
composition of matter comprises at least about 95 wt.%  
5 of one or more polyols.

9. A composition of matter comprising one  
or more zinc salts of fatty acid containing 8 to 24  
atoms, and one or more fatty alkyl diethoxylates of  
the formula  $R-N((CH_2CH_2O)_nH)_2$  wherein R is alkyl or  
10 alkenyl containing 12 to 22 carbon atoms, and n is 1  
to 5; in which said one or more fatty alkyl  
diethoxylates comprise about 1 to about 10% by weight  
of the amount of said one or more zinc salts of fatty  
acid.

15                   10. A composition of matter according to  
claim 9 wherein said one or more fatty alkyl  
diethoxylates are selected from the group consisting  
of fatty alkyl diethanolamines wherein the fatty alkyl  
substituent contains 12 to 22 carbon atoms.

20                   11. A composition of matter according to  
claim 10 wherein said one or more zinc salts of fatty  
acids comprises zinc stearate.

12. A composition of matter comprising a  
major amount of polyol and further comprising a  
25 composition according to claim 9 in an amount thereof  
such that about 0.1 to about 10 wt. % of said  
composition of matter comprises said one or more zinc  
salts of fatty acids containing 8 to 24 carbon atoms.

13. A composition of matter comprising a  
30 major amount of polyol and further comprising a  
composition according to claim 10 in an amount thereof

1 such that about 0.1 to about 10 wt. % of said  
composition of matter comprises said one or more zinc  
salts of fatty acids containing 8 to 24 carbon atoms.

14. A composition of matter according to  
5 claim 13 wherein said one or more zinc salts of fatty  
acids comprises zinc stearate.

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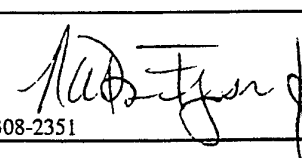
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# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US96/11713

| <b>A. CLASSIFICATION OF SUBJECT MATTER</b><br>IPC(6) :C08K 3/24, 5/16<br>US CL :252/182.26, 182.29; 524/724<br>According to International Patent Classification (IPC) or to both national classification and IPC                                                                                                                                                                                                                      |                                                                                    |                                                                                                                                                                                                                                              |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <b>B. FIELDS SEARCHED</b><br>Minimum documentation searched (classification system followed by classification symbols)<br>U.S. : 252/182.26, 182.29; 524/724<br><br>Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched<br><br>Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) |                                                                                    |                                                                                                                                                                                                                                              |
| <b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>                                                                                                                                                                                                                                                                                                                                                                                         |                                                                                    |                                                                                                                                                                                                                                              |
| Category*                                                                                                                                                                                                                                                                                                                                                                                                                             | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No.                                                                                                                                                                                                                        |
| X                                                                                                                                                                                                                                                                                                                                                                                                                                     | US, A, 4,519,965 (TAYLOR ET AL) 28 May 1985, see entire document.                  | 1-14                                                                                                                                                                                                                                         |
| X                                                                                                                                                                                                                                                                                                                                                                                                                                     | US, A, 5,155,145 (FOSTER ET AL) 13 October 1992, see entire document.              | 1-14                                                                                                                                                                                                                                         |
| <input type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.                                                                                                                                                                                                                                                                                                 |                                                                                    |                                                                                                                                                                                                                                              |
| * Special categories of cited documents:                                                                                                                                                                                                                                                                                                                                                                                              | *T                                                                                 | later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention                                              |
| *A* document defining the general state of the art which is not considered to be of particular relevance                                                                                                                                                                                                                                                                                                                              | *X*                                                                                | document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone                                                                     |
| *E* earlier document published on or after the international filing date                                                                                                                                                                                                                                                                                                                                                              | *Y*                                                                                | document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art |
| *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)                                                                                                                                                                                                                                                               | *G*                                                                                | document member of the same patent family                                                                                                                                                                                                    |
| *O* document referring to an oral disclosure, use, exhibition or other means                                                                                                                                                                                                                                                                                                                                                          |                                                                                    |                                                                                                                                                                                                                                              |
| *P* document published prior to the international filing date but later than the priority date claimed                                                                                                                                                                                                                                                                                                                                |                                                                                    |                                                                                                                                                                                                                                              |
| Date of the actual completion of the international search<br>03 SEPTEMBER 1996                                                                                                                                                                                                                                                                                                                                                        |                                                                                    | Date of mailing of the international search report<br><b>17 SEP 1996</b>                                                                                                                                                                     |
| Name and mailing address of the ISA/US<br>Commissioner of Patents and Trademarks<br>Box PCT<br>Washington, D.C. 20231<br>Facsimile No. (703) 305-3230                                                                                                                                                                                                                                                                                 |                                                                                    | Authorized officer<br>MARGARET GLASS <br>Telephone No. (703) 308-2351                                                                                   |