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STABILIZATION OF SOAP COMPOSITIONS
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This invention pertains to a method for stabilizing various cosmetic, soap, detergent and personal hygiene com- 10 positions containing certain bacteriostats and to the compositions thereby obtained.

It has been previously found by others that bacteriorstatic properties are imparted to various compositions by the incorporation therein of chlorohydroxyphenyl phenyl 15 ethers of the formula:

wherein

R is a halogen atom, and R1 is either chloro or hydrogen.

Typical bacteriostats of this type are thus 2-hydroxy-4,4' - dichlorodiphenyl ether, 2 - hydroxy - 4 - chloro - 4'bromodiphenyl ether, 2 - hydroxy - 3',4,4' - trichlorodiphenyl ether, 2 - hydroxy - 2'4,4' - trichlorodiphenyl ether and the like.

Confirming these findings, it has now been found that under certain conditions, some color formation develops in compositions containing these bacteriostats upon prolonged exposure to light. While this color formation is by no means universal, and while it is of a considerably lesser degree than that observed previous bacteriostats, it is nevertheless undesirable in colorless, white or lightly colored compositions such as soap bars, soap flakes, soap powders, shampoos, detergents, deodorants, cosmetic creams and the like.

According to the present invention, such color formation can be reduced, retarded and/or prevented by the addition to such compositions of an aromatic carboxylic acid or an alkali metal salt thereof such as the sodium or potassium salt. Such acids include particularly benzene carboxylic acids having from one to six carboxylic acid groups such as for example benzoic acid, phthalic acid, isophthalic acid, terephthalic acid, hemimellitic acid, trimellitic acid, trimesic acid, prehnitic acid, mellophanic acid, pyromellitic acid, benzenepentacarboxylic acid and mellitic acid.

One or more of such aromatic carboxylic acid or the alkali metal salt thereof is thus added to the composition containing the bacteriostat in an amount which is from 10 to 100% by weight of the amount of bacteriostat. Thus for example in a composition containing 1% of bacteriostat, the aromatic carboxylic acid will be present from about 0.1% to about 1% of the total composition.

By soaps are intended the well known surface active alkali metal salts of fatty acids. By detergents is meant both the well known nonionic surface active compounds and ionic surface active alkali metal sulfonates.

While the free acids are in practice generally used, it appears that the basic nature of the soap itself and the 10 to 15% moisture generally present in soap compositions result in conversion of the acid to the salt. Use of the acid or of its sodium or potassium salt thus are substantially equivalent embodiments of the present invention.

The color stabilizing aromatic carboxylic acid or salt 70 may be incorporated in the composition during manufacture. For example the aromatic carboxylic acid may be

added during the crutching, milling, spray drying or similar operation, either as a powder or in solution with a suitable solvent. In general any method which results in a uniform and intimate mixture may be employed. Other components normally found in such compositions such as abrasives, water softeners, perfumes, fillers and the like may be added without affecting the color stabilization realized through the present invention.

The following examples will further serve to typify the nature of this invention but are not to be construed as a limitation thereof.

Example 1

2 - amino - 4,4' - dichlorodiphenyl ether is prepared according to the method of Groves et al., J. Chem. Soc., 1929, p. 519, by treating a solution of p-chlorophenol and potassium hydroxide with 2,5-dichloronitrobenzene, followed by reduction of the product thus formed.

To a solution of 200 g. of 100% nitrosyl sulfuric acid in 1560 g. of conc. sulfuric acid are added with stirring 381 g. of 2-amino-4,4'-dichlorodiphenyl ether at a temperature of 40 to 45° C. After stirring for 3 hours at room temperature, 450 ml. of water are added with cooling. Eight hundred milliliters of o-dichlorobenzene are then added and the mixture boiled until no more diazo compound can be traced. The organic layer is separated while hot and to it are added 1000 ml. of water and 125 ml. of 30% aqueous sodium hydroxide. The mixture is steamed distilled and the residue cooled, filtered and washed. The filtrate is poured in 150 ml. of conc. hydrochloric acid and the solid which forms is collected by filtration, washed to neutrality and dried. This product is then distilled in vacuo and the distillate then recrystallized from petroleum ether to yield 2-hydroxy-4,4'dichlorodiphenyl ether, M.P. 78-79° C.

In a similar fashion by employing an equivalent amount of p-bromophenol, p-fluorophenol, 2-chlorophenol, 3,4dichlorophenol and 2,4 - dichlorophenol for p - chlorophenol in this procedure, there are respectively obtained 2 - hydroxy - 4 - chloro - 4' - bromodiphenyl ether, 2-hydroxy - 4 - chloro - 4' - fluorodiphenyl ether, 2 - hydroxy - 2',4 - dichlorodiphenyl ether, 2 - hydroxy - 3'-4,4' - trichlorodiphenyl ether and 2 - hydroxy - 2',4,4'trichlorodiphenyl ether.

Example 2

One percent by weight of 2-hydroxy-4,4'-dichlorodiphenyl ether is milled with soap and a color stabilizer added. The mixture is then exposed to approximately 400 langleys of sunlight. The following Hunter color differences (L-3b) were then recorded, the lower the value, the darker the color. Soap containing no bacteriostat gives a value of 77.0.

| Amount, percent | Stabilizer | H.C.D. |
|--|---------------|---|
| 0. 10 0. 25 0. 50 0. 50 0. 50 0. 50 0. 50 0. 50 | Phthalic acid | 36. 0 65. 1 69. 3 72. 0 72. 1 72. 1 72. 6 70. 7 72. 4 |

What is claimed is:

1. A composition comprising an alkali metal fatty acid

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soap and a germicidally effective amount of a bacteriostat of the formula:

wherein

R¹ is a member selected from the group consisting of 10 benzene carboxylic acid is isophthalic acid.

hydrogen and chloro, and about 10% to about 100% by weight of the amount of the bacteriostat of a member selected from the group consisting of a benzene carboxylic acid having from one to six carboxylic acid groups and the alkali metal salts 15

2. The composition claimed in claim 1 wherein the benzene carboxylic acid is benzoic acid.

3. The composition claimed in claim 1 wherein the benzene carboxylic acid is phthalic acid.

4. The composition claimed in claim 1 wherein the benzene carboxylic acid is isophthalic acid.

5. The composition claimed in claim 1 wherein the benzene carboxylic acid is terephthalic acid.

6. The composition claimed in claim 1 wherein the 25 benzene carboxylic acid is pyromellitic acid.

7. The composition claimed in claim 1 wherein the benzene carboxylic acid is trimesic acid.

8. A composition comprising an alkali metal fatty acid soap and a germicidally effective amount of a bacteriostat of the formula:

and about 10% to about 100% by weight of the amount

of bacteriostat of a member selected from the group consisting of a benzene carboxylic acid having from one to six carboxylic acid groups and the alkali metal salts thereof.

9. The composition claimed in claim 8 wherein the benzene carboxylic acid is benzoic acid.

10. The composition claimed in claim 8 wherein the benzene carboxylic acid is phthalic acid.

11. The composition claimed in claim 8 wherein the

12. The composition claimed in claim 8 wherein the benzene carboxylic acid is terephthalic acid.

13. The composition claimed in claim 8 wherein the benzene carboxylic acid is pyromellitic acid.

14. The composition claimed in claim 8 wherein the benzene carboxylic acid is trimesic acid.

References Cited by the Examiner

UNITED STATES PATENTS

9/1938 Evans _____ 260—611.5 2,130,080 Mitoray et al. ____ 252—107 3,085,066 4/1963

FOREIGN PATENTS

8/1964 Netherlands. 6,401,526

OTHER REFERENCES

Chemical Abstracts, vol. 51, p. 11300i.

Organic Chemistry, Fieser et al., D. C. Heath and Co., Boston (1944), p. 679.

Soap and Sanitary Chemicals, "Stabilization of White Milled Soaps," Fiore (December 1941) pp. 30-33, 75

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