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DISINFECTANTS

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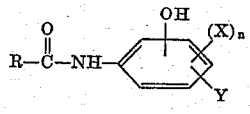
The present invention relates to disinfectants, and more particularly to germicidal acylated aminophenols and to compositions comprising such germicidal agents.

I have found that acylated aminophenols having the structural formula



wherein R is alkenyl with 5 to 20 carbon atoms and from 1 to 3 olefinic bonds, X is chlorine or bromine, *n* is an integer from 1 to 3, inclusive, and Y is hydrogen, chlorine or bromine, are powerful germicidal agents, and that compositions having said acylated aminophenols incorporated therein posses excellent disinfectant properties.

The novel compounds according to the present invention are soluble in water and have properties similar to soap. In other words, they form foaming solutions and may therefore be combined with other soaps without detriment to the characteristic effects of either soapy component.

The presence of olefinic bonds in the fatty acid moiety of acylated aminophenols is of considerable importance to their germicidal activity. Thus, saturation of the olefinic bonds leads to a substantial loss in germicidal activity, as illustrated by the following table:

TABLE

Acylated Aminophenol	Dilution	Growth of *Staphylococcus Aureus*	Growth of Coliform Bacteria
Undecylenic acid-2-hydroxy-5-chloro-anilide	1:2,100,000	−	
	1:5,100,000	+	
Sorbic acid-2-hydroxy-5-chloro-anilide	1:51,000	−	
	1:110,000	+	
	1:45,000		−
	1:81,000		+
Undecylic acid-2-hydroxy-5-chloro-anilide	1:21,000	−	
	1:51,000	+	
	1:1,100		−
	1:2,100		(+)
	1:5,100		+

In the above table

− indicates that no bacteria growth was observed;
+ indicates that definite bacteria growth was observed;
(+) indicates that an insignificant amount of bacteria growth was observed.

Evaluation of the values tabulated above clearly shows that very dilute solutions containing as little as 1 part per 2 million of an acylated aminophenol having double bonds in the fatty acid moiety are effective in inhibiting the growth of *Staphylococcus aureus*, whereas much less dilute solutions of an acylated aminophenol having only saturated bonds in the fatty acid moiety are ineffective. The greater germicidal activity of sorbic acid-2-hydroxy-5-chloro-anilide as compared to the corresponding undecylic acid anilide with respect to coliform bacteria is also evident from the tabulated values.

The acylated aminophenols according to the present invention may be incorporated into body powders, after-shave lotions, bath water additives, cleansing agents for use in dairies, hospitals and the like, dish-rinsing compositions and all other compositions and solutions intended for use in suppressing the growth and propagation of bacteria, in concentrations between 0.1 and 8.0% by weight, and preferably 1 to 3% by weight.

The following examples are given to illustrate various germicidal compositions containing the acylated aminophenols according to the present invention which may be compounded:

Example I

BODY POWDERS

A

	Gm.
Starch	200
Zinc oxide	100
Talcum	150
Magnesium carbonate	20
Titanium oxide	20
Acylated aminophenol	10

B

Talcum	340
Kaolin	60
Zinc oxide	100
Magnesium carbonate	10
Titanium oxide	20
Acylated aminophenol	10

Example II

AFTER-SHAVE LOTION

	Gm.
Water	750
Alcohol	250
Menthol	1.5
Lemon oil	2.0
Orange oil	1.0
Neroli oil	0.5
Lavender oil	0.5
Acylated aminophenol	10.0

Example III

DISH-RINSING COMPOSITION

	Gm.
Trisodium phosphate	60
Tetrasodium pyrophosphate	30
Alcohol sulfonate	5
Acylated aminophenol	5

Example IV

CLEANSING COMPOSITION FOR USE IN DAIRIES

	Gm.
Soda	45
Trisodium phosphate	35
Sodium silicate	10
Caustic soda	5
Acylated aminophenol	5

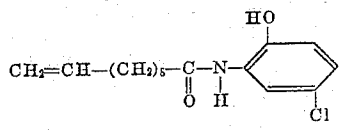
In Examples I through IV above the term "acylated aminophenol" is intended to mean any acylated aminophenol containing at least one olefinic bond in the fatty acid moiety and having the structural formula defined above. In addition to the acylated aminophenols according to the present invention, other germicidal agents may of course also be included in the compositions illustrated in Examples I to IV, if desired.

The olefinic acylated aminophenols according to the present invention are advantageously produced by react-

ing an olefinic acid chloride with a halogenated aminophenol, as illustrated by the following examples:

Example V

UNDECYLENIC ACID-2-HYDROXY-5-CHLORO-ANILIDE

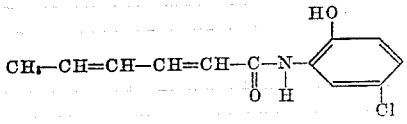


4.7 gm. undecylenic acid chloride and 12.2 gm. 2-amino-4-chlorophenol were dissolved in dioxan, and the solution was heated for 4 hours at 35–40° C. accompanied by stirring. The reaction mixture was then allowed to stand overnight, whereupon the solvent was removed by vacuum distillation. The distillation residue was washed several times with 2 N hydrochloric acid and was then recrystallized from alcohol in the presence of animal charcoal. Colorless crystals having a melting point of 80–81° C. were obtained. The yield was 5.5 gm., which corresponds to 76.6% of the theoretical yield.

$C_{17}H_{24}ClNO_2$.—Calculated: C, 65.89; H, 7.81; N, 4.52%. Found: C, 66.02; H, 7.94; N, 4.86%.

Example VI

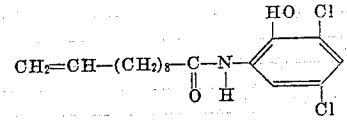
SORBIC ACID-2-HYDROXY-5-CHLORO-ANILIDE



By a procedure analogous to that described in Example V, 2.0 gm. sorbic acid-2-hydroxy-5-chloro-anilide were obtained from 2.5 gm. sorbic acid chloride and 4.7 gm. 2-amino-4-chlorophenol. The yield corresponded to 50% of the theoretical yield. The product was a colorless crystalline substance having a melting point of 172–173° C.

Example VII

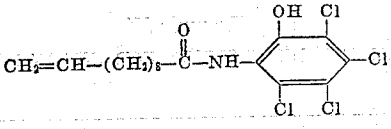
UNDECYLENIC ACID-2-HYDROXY-3,5-DICHLORO-ANILIDE



Following the procedure described in Example V, undecylenic acid-2-hydroxy-3,5-dichloro-anilide was obtained from undecylenic acid chloride and 2-amino-4,6-dichlorophenol.

Example VIII

UNDECYLENIC ACID-2-HYDROXY-3,4,5,6-TETRACHLORO-ANILIDE

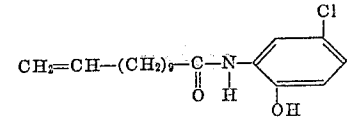


4.5 gm. undecylenic acid chloride were added dropwise to a solution of 12.7 gm. 2-amino-3,4,5,6-tetrachlorophenol in dioxan under anhydrous conditions. The resulting mixture was allowed to stand overnight and was then heated for 6 hours at 40–45° C. accompanied by stirring. Thereafter, the dioxan solvent was removed by vacuum distillation and the residue was recrystallized several times from ethanol in the presence of animal charcoal. The recrystallized product was obtained in the form of colorless needles having a melting point of 108–109° C. The yield was 7.4 gm., which corresponds to 73% of the theoretical yield.

$C_{17}H_{21}Cl_4NO_2$.—Calculated: C, 49.72; H, 5.12; N, 3.39%. Found: C, 49.56; H, 5.13; N, 3.67%.

Example IX

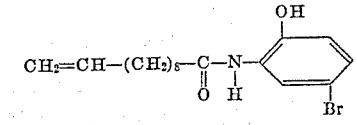
DODECYLENIC ACID-Δ^{11}-2-HYDROXY-5-CHLORO-ANILIDE



6 gm. dodecylenic acid chloride were added dropwise, at room temperature, to a mixture of 4 gm. 4-chloro-2-aminophenol, 150 cc. anhydrous ether, 50 cc. dioxan and 2.5 cc. pyridine, accompanied by vigorous mechanical stirring. The resulting mixture was allowed to stand overnight and was then stirred for an additional six hours at 40° C. on a water bath. Thereafter, the ether was removed by vacuum distillation and the residue was treated with warm 2 N hydrochloric acid to remove the pyridine-chlorohydrate formed by the reaction. The resulting mixture was then filtered and the filter cake was recrystallized from ethanol. The yield was 3.1 gm. The crystalline product had a melting point of 88–90° C.

Example X

UNDECYLENIC ACID-2-HYDROXY-5-BROMO-ANILIDE

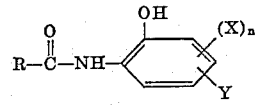


4.7 gm. undecylenic acid chloride and 15 gm. 2-amino-4-bromophenol were dissolved in dioxan, and the solution was heated for 4 hours at 35–40° C. accompanied by stirring. The reaction mixture was then allowed to stand overnight, whereupon the solvent was removed by vacuum distillation. The distillation residue was washed several times with 2 N hydrochloric acid and was then recrystallized from ether/water. Colorless crystals having a melting point of 76° C. Yield 68% of the theoretical yield.

While I have illustrated certain specific embodiments of the present invention, it will be readily apparent to those skilled in the art that the invention is not limited to these embodiments, and that various changes and modifications may be made without departing from the spirit of the invention or the scope of the appended claims.

I claim:

1. Acylated aminophenols having the structural formula



wherein R is alkenyl with 5 to 20 carbon atoms having from 1 to 3 olefinic double bonds, X is selected from chlorine and bromine, *n* is an integer from 1 to 3, inclusive, and Y is selected from the group consisting of hydrogen, chlorine and bromine.

2. Undecylenic acid-2-hydroxy-5-chloro-anilide.

3. Sorbic acid-2-hydroxy-5-chloro-anilide.

4. Undecylenic acid-2-hydroxy-3,5-dichloro-anilide.

5. Undecylenic acid-2-hydroxy-3,4,5,6-tetrachloro-anilide.

6. Dodecylenic acid-Δ''-2-hydroxy-5-chloro-anilide.

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