

UNITED STATES PATENT OFFICE

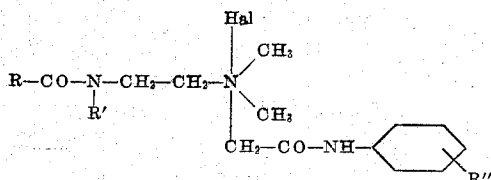
2,644,003

QUATERNARY AMMONIUM COMPOUNDS
AND THEIR MANUFACTUREHans Gysin and Rudolf Edward Hagenbach,
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A. G., Basel, Switzerland, a Swiss firmNo Drawing. Application February 2, 1951, Serial
No. 209,210. In Switzerland February 24, 1950

5 Claims. (Cl. 260—404.5)

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According to the present invention, new quaternary ammonium compounds are provided having the general formula:



wherein R represents an alkyl radical with 7-15 C atoms,

Hal represents chlorine or bromine,

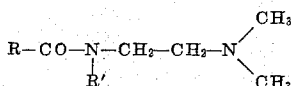
R' represents a low alkyl radical, and

R'' represents hydrogen, chlorine, bromine, an alkyl or alkoxy radical, or more of these substituents.

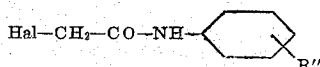
It has been found that these compounds are distinguished from the previously known quaternary ammonium compounds by a very strong bactericidal activity and at the same time in contrast to the greater part of the latter compounds, the compounds according to the present invention also have the advantage that they are mostly solid crystallised bodies and therefore come into consideration for a greater number of intended uses.

The new compounds can be produced by various related processes.

The basic amides of the general formula:

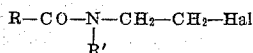


can be reacted with halogen acetanilides of the general formula:

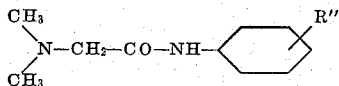


In these and all following formulae, R, Hal, R' and R'' always have the meanings given above.

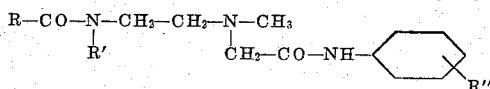
Also, compounds of the general formula:



can be reacted with dimethylamino acetanilides of the general formula:



Further, compounds of the general formula:



can be reacted in the autoclave with methyl chloride or methyl bromide.

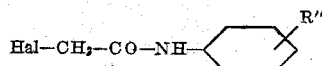
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The above reactions may take place in the presence or absence of suitable solvents such as benzene, toluene, xylene, acetone, ethyl and butyl acetate and also water, and may be simplified if required by the addition of small amounts of potassium iodide.

The basic amides used in the first above reaction can be obtained for example by heating higher fatty acids, in particular lauric acid or myristic acid, or also palmitic acid, with low dimethylamino ethyl alkylamines for a fairly long period. Derivatives of the free fatty acids, in particular acid halides or esters, may also be used.

In this reaction it is not necessary that the alkyl radical of the fatty acid should have a homogenous chain. Mixtures of higher fatty acids may be used with advantage, as they result from the saponification of vegetable and animal fats and oils. Such mixtures of fatty acids, e. g. from palm kernel fats, apart from fatty acids with an average alkyl chain length of 11-13 carbon atoms, also contain small portions of lower and higher fatty acids. By distilling once, these portions can be separated and then a fatty acid mixture is obtained with an alkyl chain length of substantially 11-13 carbon atoms. The radical of this fatty acid mixture is referred to in the examples as "Lauroyl" radical.

Chloraceto- and bromaceto anilide, -o-toluidide, -m-toluidide, -p-toluidide, -o-anisidide, -m-anisidide, -p-anisidide, -o-chloranilide, -m-chloranilide, -p-chloranilide, -p-bromanilide, -3,4-dichloranilide, -2,4-dimethylanilide and -2,5-dimethylanilide can be named as examples of halogen acetanilides of the general formula:



used according to the present invention.

As aqueous solutions, the new compounds can be used for the disinfection of human and animal skin, and also for disinfection of rooms, floors, or utensils of all kinds such as cutlery, crockery, medical instruments, patients' personal and bed linen. The solutions for use may be prepared in the usual way, e. g. by diluting concentrated solutions or also by direct dissolution of the solid compounds in water. For this latter purpose the new compounds may also be prepared in tablet form. Further, they can also be adapted for various intended uses by the addition of other materials with a disinfectant action, such as phenols, or of rust-proofing agents such as sodium nitrite or sodium salt of octyloxyacetic acid. They can also be combined with pulverulent substances or with organic solvents and also incorporated in ointments and pastes. The disinfectant action is excellent both against gram positive and gram negative germs such as staphylococci, and bacterium coli, typhus, paratyphus B, bacterium pyocyaneum, enteritis Gärtner, etc. respectively.

As a result of the capillary active properties of the new compounds, they also come into consideration as wetting and dispersing agents for various technical purposes, for example in the leather, paper and textile industries.

The following examples serve to illustrate the invention. Parts are always given as parts by weight and temperatures are in degrees centigrade.

Example 1

284 parts of dodecanoyl-N,N-dimethylamino ethyl-N'-methyl amide (boiling point 140-160° at 0.3 mm. pressure) produced, for example from lauric acid and trimethyl ethylenediamine, are boiled for 6 hours under reflux with 169.5 parts of chloracetanilide in 800 parts of ethyl acetate. The precipitate which forms on standing is drawn off by suction. (Dodecanoyl-N'-methyl-amino-ethyl) - (phenylcarbamy-methyl) - dimethyl ammonium chloride is obtained on recrystallization from acetone as a white substance. It melts at 124° and is completely soluble in water.

The corresponding bromide is produced in an analogous manner.

Example 2

298 parts of dodecanoyl-N,N-dimethylamino-ethyl-N'-ethylamide and 183.5 parts of chlor-aceto-p-toluidide are heated for 8 hours under reflux with 700 parts of benzene. The benzene is distilled off and the solid residue is recrystallized from acetone. (Dodecanoyl-N'-ethyl-aminoethyl) - (p-tolylcarbamy-methyl) - dimethyl ammonium chloride is obtained as white crystals. It melts at 182° and dissolves completely in water.

The corresponding bromide is produced in an analogous manner.

Example 3

312 parts of dodecanoyl-N,N-dimethylamino-ethyl-N'-propylamide (B. P. 130-150° at 0.3 mm. pressure), prepared for example from lauric acid methylester and N,N-dimethyl-N'-propyl-ethylenediamine, are heated for 12 hours in a boiling water bath while stirring with 169.5 parts of chloracetanilide. After cooling, the solid mass is recrystallized from ethyl acetate/acetone. (Dodecanoyl - N'-propylamino ethyl) - (phenylcarbamy-methyl) - dimethyl ammonium chloride is obtained as a white crystalline substance. It melts at 135° and is completely soluble in water.

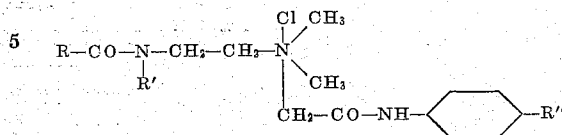
The corresponding bromide is produced in an analogous manner.

The following compounds may also be prepared in the above described manner:

- (Dodecanoyl-N'-methylamino ethyl) - (p-chloro-phenylcarbamy-methyl) - dimethyl ammonium chloride, M. P. 116°.
- (Dodecanoyl - N'-ethylamino ethyl) - (phenylcarbamy-methyl) - dimethyl ammonium chloride. M. P. 164°.
- (Dodecanoyl-N'-propylamino ethyl) - (p-tolylcarbamy-methyl) - dimethyl ammonium chloride. M. P. 144°.
- ("Lauroyl"-N'-methylamino ethyl) - (phenylcarbamy-methyl) - dimethyl ammonium chloride. M. P. about 110°.
- (Dodecanoyl - N' - methylaminoethyl) - (p-methoxyphenylcarbamy-methyl) - dimethyl ammo-

What we claim is:

1. A quaternary ammonium chloride of the formula:



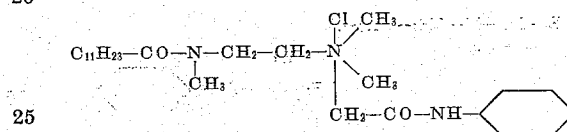
10 wherein

R represents an alkyl chain of from 9 to 15 carbon atoms,

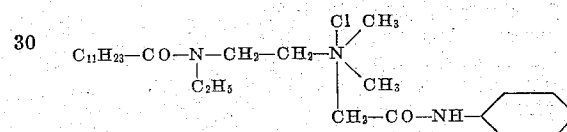
R' represents an alkyl radical of from 1 to 3 carbon atoms,

15 R'' represents a substituent selected from the group consisting of hydrogen, chlorine, methyl and methoxy.

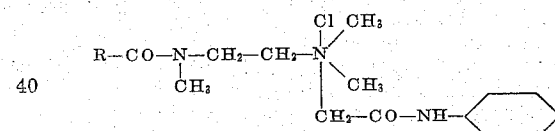
2. A quaternary ammonium chloride of the formula:



3. A quaternary ammonium chloride of the formula:

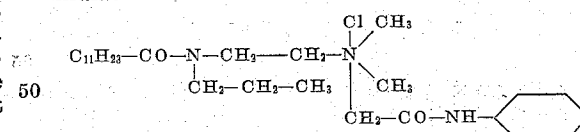


35 4. A quaternary ammonium chloride of the formula:



wherein R represents an alkyl chain of from 11 to 13 carbon atoms.

45 5. A quaternary ammonium chloride of the formula:



HANS GYSIN.

RUDOLF EDWARD HAGENBACH.

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