

UNITED STATES PATENT OFFICE

2,459,062

QUATERNARY AMMONIUM COMPOUNDS

Elmer W. Cook, New York, N. Y., and Philip H. Moss, Greenwich, Conn., assignors to American Cyanamid Company, New York, N. Y., a corporation of Maine

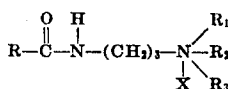
No Drawing. Application February 8, 1944,
Serial No. 521,588

5 Claims. (Cl. 260-404.5)

1

The present invention is concerned with the production of new organic ammonium compounds which are adapted for various uses, more particularly as germicidal agents, wetting agents, and softening agents for textiles and the like.

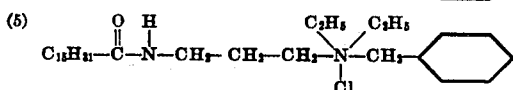
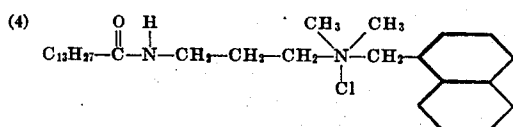
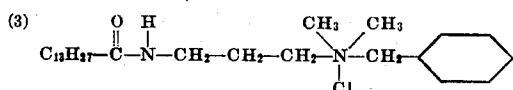
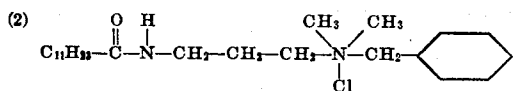
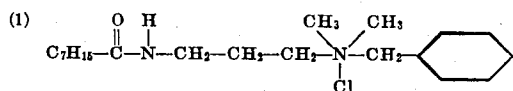
These compounds are of the quaternary ammonium type which conform to the general formula—



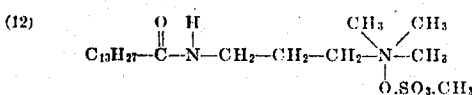
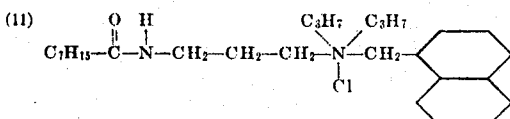
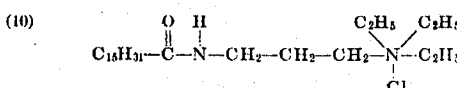
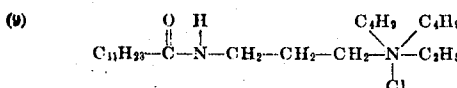
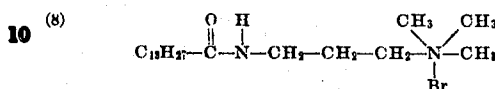
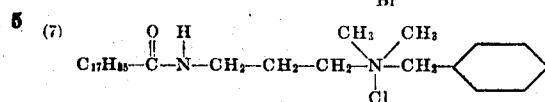
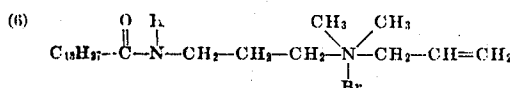
in which R is an aliphatic hydrocarbon radical containing at least seven carbon atoms, **R₁ and R₂** represent alkyl groups of low molecular weight such as for example methyl, ethyl, propyl, isopropyl, butyl, etc., **R₃** represents a member of the group consisting of alkyl, aralkyl and unsaturated aliphatic hydrocarbon radicals, and **X** represents an anion such as, for example, a halide, hydrogen sulfate, methyl or ethyl sulfate, alkyl phosphate, thiocyanate, etc.

The new products are in part crystalline, in part thickly liquid, viscous substances which in general dissolve readily in water and form stable aqueous solutions. In view of their dispersing and disinfecting properties, they exert a remarkable action as wetting and emulsifying agents and also as antiseptics. They are practically odorless and relatively non-toxic to man.

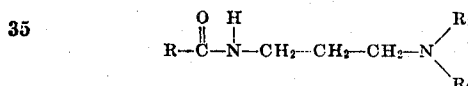
Representative compounds which fall within the scope of this invention are:



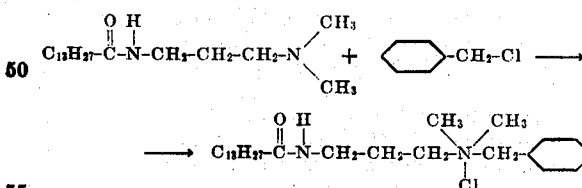
2



These quaternary ammonium salts may be prepared by treating a tertiary amine of the formula—

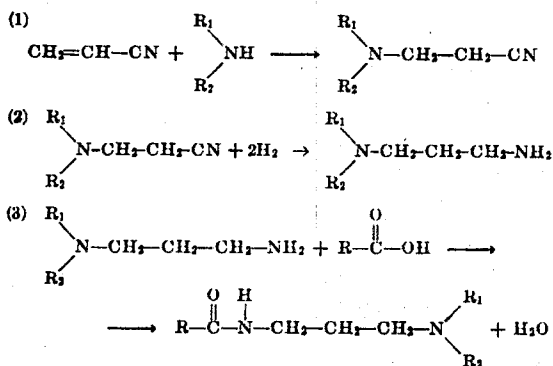


in which R, **R₁** and **R₂** represent the molecular groups above referred to, with an alkylating agent such as an alkyl halide, dialkyl sulfate, aralkyl chloride, etc. Thus, for example, γ -myristamidopropyldimethylamine treated with benzyl chloride produces the ammonium salt, γ -myristamidopropyldimethylbenzylammonium chloride according to the reaction



3

Reactions which may be involved in the preparation of the tertiary amine may be outlined as follows:



As an alternative method for step (3), the acid chloride,



may be used in place of the acid.

The invention is further illustrated by the following examples, the parts being by weight unless otherwise stated.

Example I

Into 545 parts of a 25% aqueous solution of dimethyl amine, cooled with ice and water, there were added from a dropping funnel 170 parts of acrylonitrile. The rate of addition of the nitrile was controlled so that the temperature in the reaction vessel remained below 20° C. After the cold reaction mixture had stood for one hour, it was poured into 350 cc. of 10% aqueous sodium hydroxide solution, the oily layer collected and the aqueous layer extracted with ether. The extract and the oily layer were dried over sodium sulfate and then distilled. 218 parts of β -dimethylaminopropionitrile were collected at 73-74° C./22 mm.

207 parts of β -dimethylaminopropionitrile were hydrogenated in an autoclave under a pressure of about 90 atmospheres at 100° C. in the presence of 72.4 parts of anhydrous ammonia, using Raney nickel as the catalyst. The product was dried over solid potassium hydroxide and vacuum distilled. 204.5 parts of N,N-dimethylpropylenediamine were collected, B. P. 134° C. at atmospheric pressure.

38 parts of myristyl chloride were added dropwise to a solution of 15.5 parts of N,N-dimethylpropylenediamine in 160 parts of benzene. After one hour of stirring, the benzene solution was washed with 10% aqueous sodium hydroxide. The benzene layer was then washed once with water and the solvent removed by vacuum distillation. The residue was distilled at 208-215° C./1-2 mm., giving a solid distillate of γ -myristamidopropyl dimethylamine.

A solution of 6.2 parts of γ -myristamidopropyl dimethylamine and 3.4 parts of benzyl chloride in 30 parts of benzene was refluxed during a period of four hours. The benzene was removed under vacuum, giving a light-colored, amorphous semi-solid at room temperature, which melts at about 54° C. to a clear, straw-colored liquid. This quaternary ammonium salt, γ -myristamidopropyl dimethylbenzylammonium chloride, is soluble in most organic solvents and forms a clear 25% solution in water.

This compound is an excellent germicidal

4

agent. It has been found effective against *Staphylococcus aureus* in a dilution of one part in 25,000 at 37° C. during a test of five minutes. The phenol coefficient of the compound is in the range of 277 to 333.

This product is also particularly suitable as a wetting agent for cotton fabrics.

Example II

A mixture consisting of 30 parts of benzene, 8.1 parts of γ -caprylamidopropyl dimethylamine and 5 parts of benzyl chloride was heated at reflux temperature for six hours. After removing the benzene, the γ -caprylamidopropyl dimethylbenzylammonium chloride was obtained as a viscous syrup.

Example III

Two parts of benzyl chloride were gradually added to a solution of 4.5 parts of γ -palmitamidopropyl dimethylamine dissolved in 30 parts of benzene. The resulting solution was refluxed for about four hours. The benzene was removed by vacuum distillation, leaving the quaternary ammonium salt, γ -palmitamidopropyl dimethylbenzylammonium chloride, as an opaque solid, readily soluble in water. This product is an effective germicide, and also a good softening and wetting agent for textile fabrics.

Example IV

A solution of 6.9 parts of γ -lauramidopropyl dimethylamine and 4.5 parts of benzyl chloride in 75 parts of benzene was refluxed during a period of four hours. After removing the solvent, the γ -lauramidopropyl dimethylbenzylammonium chloride was obtained as a colorless viscous mass, soluble in water. This product is a good germicide, and may be advantageously used for various disinfecting purposes.

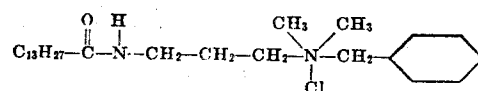
Other quaternary ammonium salts can be prepared according to the procedure followed in the above examples, such as the γ -myristamidopropyl dimethyl - α -naphthylmethylammonium chloride, γ -oleamidopropyl dimethylbenzylammonium chloride, γ -lauramidopropyl diethylmethylammonium bromide, γ -stearamidopropyl dimethylbenzylammonium chloride and γ -myristamidopropyl dimethylallylammonium bromide.

The γ -stearamidopropyl dimethylbenzylammonium chloride is an excellent softening agent for rayon and cotton fibers, and acts as a permanent re-wetting agent on fabrics.

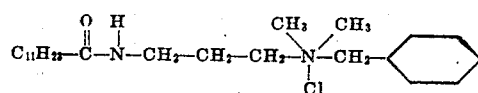
While the invention has been described with particular reference to specific embodiments, it is to be understood that it is not to be limited thereto but is to be construed broadly and restricted solely by the scope of the appended claims.

We claim:

1. A germicidal composition containing as an effective germicidal agent the quaternary ammonium compound of the following formula

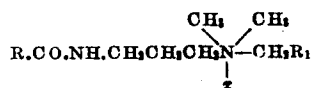


2. A germicidal composition containing as an effective germicidal agent the quaternary ammonium compound of the following formula



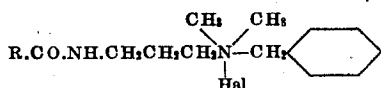
5

3. A germicidal composition containing as an effective germicidal agent a compound of the formula



in which R is an alkyl radical of 11-13 carbon atoms inclusive, R₁ is a member of the group consisting of hydrogen, methyl and phenyl, and x is a halogen.

4. A germicidal composition containing as an effective germicidal agent a compound of the formula

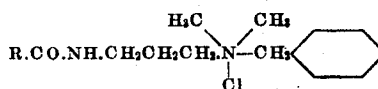


in which R is an alkyl radical of 11-13 carbon atoms inclusive and Hal is a halogen.

5. A germicidal composition comprising, as an

6

effective germicidal agent, an aqueous solution of a compound of the formula



in which R is an alkyl radical of 11-13 carbon atoms.

ELMER W. COOK.
PHILIP H. MOSS.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,860,563	Wayne	May 31, 1932
1,737,458	Hartmann et al.	Nov. 26, 1939
2,262,741	De Groote	Nov. 11, 1941
2,345,570	Bley	Apr. 4, 1944
2,362,760	Maxwell	Nov. 14, 1944