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QUATERNARY AMMONIUM PHOSPHITES

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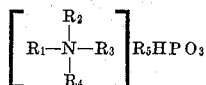
This invention relates to new organic ammonium phosphites and relates more particularly to quaternary ammonium phosphites which are suitable for various uses as wetting agents, detergents, emulsifying agents, germicides, fungicides, textile softeners, textile lubricants, dyeing assistants, anti-static agents, textile spinning bath assistants, vinyl resin stabilizers, lubricant additives, etc.

It is an object of the present invention to provide a new group of quaternary ammonium hydrogen phosphite compounds.

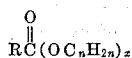
It is another object of this invention to provide for a method of manufacturing the said quaternary ammonium hydrogen phosphite compounds.

These and other objects and advantages will become obvious to those skilled in the art from the following disclosure.

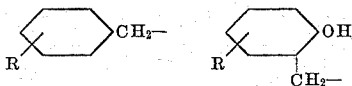
The compounds of this invention are defined by the formula:



R_1 is an organic radical containing as its main component a lipophilic radical that contains from 8 to 20 carbon atoms. This radical may be present as an aliphatic hydrocarbon radical. In this form it contains from 10 to 20 carbon atoms. It may be present in the form



wherein R is an aliphatic hydrocarbon radical containing from 10 to 20 carbon atoms, n is 2 to 3 and x is 1 to 10. Such a group is usually obtained by treating a suitable secondary amine with an alkylene oxide and then esterifying the reaction product with a fatty acid containing from 10 to 20 carbon atoms. The lipophilic radical may be present as a substituent in a substituted benzyl radical, i. e.,



etc., in which case R contains from 8 to 16 carbon atoms.

R_2 , R_3 , R_4 and R_5 are lower aliphatic hydrocarbon radicals. It is preferred that R_2 and R_3 be methyl groups.

These products are formed by the reaction of a dialkyl hydrogen phosphite and a tertiary amine. Suitable dialkyl hydrogen phosphites are di-methyl hydrogen phosphite, di-ethyl hydrogen phosphite, di-butyl hydrogen phosphite, methyl ethyl hydrogen phosphite, ethyl butyl hydrogen phosphite, etc. The tertiary amines adapted to the purposes of this invention may be represented by the generic formula:



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wherein R'_1 is a monovalent lipophilic organic radical, R'_2 and R'_3 are lower alkyl groups.

Suitable tertiary amines include (but are not limited to) lauryl dimethylamine, cetyl dimethylamine, octadecyl dimethylamine, dimethyl amino ethyl stearate, dimethyl amino ethyl oleate, 2-hydroxy-5-nonyl benzyl dimethylamine, dodecyl benzyl dimethylamine, tetradecyl dimethylamine, tetradecyl methyl ethyl amine, cetyl diethylamine, cetyl methyl ethylamine, dimethyl amino ethyl laurate, dimethyl amino ethyl myristate, dimethyl amino ethyl palmitate, dimethyl amino propyl myristate, diethyl amino ethyl palmitate, diethyl amino ethyl myristate, octadecenyl dimethyl amine, the laurate of polyoxyethylene dimethylamine, the oleate of polyoxyethylene dimethylamine, etc. The length of the polyoxyethylene chain in this latter type of compound may be from 1 to 10.

The dialkyl hydrogen phosphite is reacted with a suitable tertiary amine by heating at a temperature which may range from 80 to 150° C. The preferred reaction temperature is from 100 to 130° C. The reaction time will usually vary depending on the dialkyl hydrogen phosphite used. In this respect the di-methyl hydrogen phosphite appears more reactive than other di-alkyl hydrogen phosphites and less reaction time is required.

It has been observed that the compounds of the present invention possess a very unique property in that they appear to readily absorb oxygen from the air. This property indicates that these quaternary ammonium hydrogen phosphites are valuable reducing agents.

The following examples are given by way of illustration and are not to be taken as limiting the invention in any way.

Example 1

30 gms. of octadecyl dimethyl amine and 14 gms. of di-ethyl hydrogen phosphite were placed in a flask, then heated with stirring over a steam bath at a temperature of 95° C. for 60 hours. The reaction product was a water soluble low melting solid.

Example 2

150 gms. of octadecyl dimethyl amine and 70 gms. of di-ethyl hydrogen phosphite were placed in a flask, then heated with stirring over a steam bath at a temperature of 95° C. for a period of 95 hours. The reaction product was a light colored water soluble solid with a low melting point.

Example 3

102 gms. of lauryl dimethyl amine and 93 gms. of di-butyl hydrogen phosphite were placed in a flask and heated with stirring at a temperature of 95° C. for a period of 120 hrs. The product was a water soluble viscous liquid.

Example 4

600 gms. of octadecyl dimethyl amine and 280 gms. of di-ethyl hydrogen phosphite were placed in a flask and heated at a temperature ranging from 110 to 118° C. for a period of 16 hours. The product was a light colored water soluble low melting solid.

Example 5

300 gms. of octadecyl dimethyl amine and 70 gms. of di-ethyl hydrogen phosphite were placed in a flask and heated with stirring at a temperature ranging from 100 to 122° C. for a period of 17 hours. The product was a low melting solid which was water soluble.

Example 6

200 gms. of di-methyl amino ethyl stearate and 86 gms. of di-ethyl hydrogen phosphite were placed in a

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flask and heated at a temperature ranging from 107 to 120° C. for a period of 12½ hours, to yield a product that was a water soluble low melting solid.

Example 7

364 gms. of octadecyl dimethyl amine and 135 gms. of di-methyl hydrogen phosphite were placed in a flask. They were not miscible at room temperature. The mixture was heated on a steam bath, shaking from time to time. After a few minutes heating the two reactants were miscible. The reaction mixture began to solidify on the steam bath after about 50 minutes heating and after 80 minutes it appeared to be completely solid. The mixture was heated on the steam bath for a total of 8½ hours. The product was a rather hard waxy solid which was water soluble, giving clear foaming solutions.

Example 8

71 gms. of dimethyl amino ethyl stearate and 22 gms. of dimethyl hydrogen phosphite were heated together on the steam bath for 21½ hours at a temperature of 95° C. The product was a stiff paste when hot and a waxy solid when cold. It was water soluble giving clear foaming solutions.

Example 9

32 gms. of 2-hydroxy-5-dodecyl benzyl dimethyl amine and 11 gms. of dimethyl hydrogen phosphite were heated together at 100° C. The reaction became exothermic and the temperature rose to 130° C. When the exothermic reaction had subsided, the viscous reaction mixture was heated in an oil bath at 110° C. for another hour to complete the reaction. The resinous product was water dispersible, giving opaque foaming solutions.

Example 10

114 gms. of cetyl dimethyl amine and 44.7 gms of dimethyl hydrogen phosphite were heated together with stirring on a steam bath. After 30 minutes of heating the reaction mass began to crystallize and after 1 hour it was a very thick crystalline slurry. After 2 hours the reaction mass was almost non-stirrable. When cooled to room temperature, it was a wax-like product which was water soluble giving clear foaming aqueous solutions.

In the preparation of the compounds elected by the preceding examples, it has been found desirable to blanket the reaction with an inert gas or to carry out the reaction in a closed vessel, thus reducing the opportunity for oxygen absorption by these compounds.

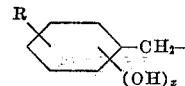
It will be understood that the embodiments of the invention described in the specification and illustrated by the examples are only illustrative of the compounds and the manner in which they are produced. Various modifications can be made without departing from the principles of the invention.

What is claimed is:

1. A quaternary ammonium alkyl hydrogen phosphite wherein the said alkyl is lower alkyl, wherein one of the radicals attached to the quaternary nitrogen atom is a monovalent lipophilic organic radical selected from the group consisting of alkyl radicals containing from 10 to 20 carbon atoms, fatty-acyl radicals containing from 11

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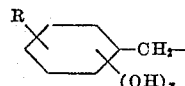
to 21 carbon atoms in the acyl residue and from 1 to 10 oxy-alkylene groups of 2 to 3 carbon atoms each, and substituted benzyl radicals conforming to the formula



wherein R represents an alkyl radical containing from 8 to 16 carbon atoms and x is from 0 to 1, and wherein the remaining 3 radicals attached to the quaternary nitrogen atom are lower alkyl.

2. A quaternary ammonium compound as in claim 1 wherein the said lipophilic organic radical is an alkyl radical containing from 10 to 20 carbon atoms.

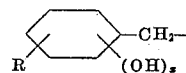
3. A quaternary ammonium compound as in claim 1 wherein the said lipophilic radical is a substituted benzyl radical conforming to the formula



wherein R is an alkyl radical containing from 8 to 16 carbon atoms and x is from 0 to 1.

4. A quaternary ammonium compound as in claim 1 wherein the said lipophilic organic radical is a fatty-acyl polyoxalkylene radical containing from 11 to 21 carbon atoms in the acyl residue and containing from 1 to 10 oxyalkylene groups of 2 to 3 carbon atoms each.

5. The process which comprises heating a tertiary amine containing, attached to the nitrogen, two lower alkyl radicals and a monovalent lipophilic organic radical selected from the group consisting of alkyl radicals containing from 10 to 20 carbon atoms, fatty-acyl polyalkylene radicals containing from 11 to 21 carbon atoms in the acyl residue and from 1 to 10 oxy-alkylene groups of 2 to 3 carbon atoms each, and substituted benzyl radicals conforming to the formula



wherein R represents an alkyl radical containing from 8 to 16 carbon atoms and x is from 0 to 1 with a di(lower) alkyl ester of phosphorous acid at a temperature of 80° to 150° C., to form a quaternary ammonium alkyl hydrogen phosphite.

6. Ethyl dimethyl octadecyl ammonium ethyl hydrogen phosphite.

7. Ethyl dimethyl cetyl ammonium ethyl hydrogen phosphite.

8. The stearate of ethyl dimethyl hydroxy ethyl ammonium ethyl hydrogen phosphite.

9. The oleate of ethyl dimethyl hydroxy ethyl ammonium ethyl hydrogen phosphite.

10. The process as defined in claim 5 wherein the said ester is diethyl hydrogen phosphite.

References Cited in the file of this patent

UNITED STATES PATENTS

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