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ACETONYL ALKYLBENZYL DIALKYL AMMONIUM SALTS

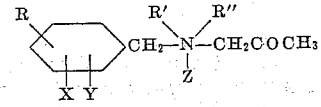
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8 Claims. (Cl. 260—567.6)

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This invention relates to quaternary ammonium salts of the structure



wherein R is an alkyl group of eight to sixteen carbon atoms, X and Y are hydrogen or methyl groups, Z is a salt-forming anion, and R' and R'' are methyl or ethyl groups. These compounds show good bactericidal action coupled with good solubility and stability.

The preferred method of making these compounds comprises reacting together in about molecular proportions an alkylbenzyl halide in which the alkyl group contains eight to sixteen carbon atoms and a dialkylaminoacetone in which the alkyl group contains not over two carbon atoms.

As is known, dimethylamine or diethylamine react with chloroacetone to give

$$R'R''NCH_2COCH_3$$

The same end result is achieved by hydrating dimethylaminopropyne or diethylaminopropyne (cf. J. Chem. Soc. 1949, 782). We have discovered that reaction of the above dialkylaminoacetones with alkylbenzyl halides yields valuable quaternary ammonium salts which are characterized by an unusual combination of properties.

Suitable alkylbenzyl halides which include also alkylmethylbenzyl and alkyldimethylbenzyl halides are available through the halomethylation of alkylbenzenes in which the alkyl group contains eight to sixteen carbon atoms and preferably nine to sixteen carbon atoms. The alkyl group can be of straight or branched chain structure. Particularly useful and economical are alkyl groups obtained from propylene polymers, fractions of which can be taken to give groups predominantly of 9, 12, or 15 carbon atoms. Another economical source of a long-chained alkyl group is from close-cut fractions of kerosene, a fraction having an average size of 14 carbon atoms being quite desirable.

Useful starting hydrocarbons are alkylated benzenes, toluenes, and xylenes, which include n-octylbenzene, n-octyltoluene, n-octylxylenes, 2-ethylhexylbenzene, 1-methylheptyltoluene, (1,3-dimethyl-1-propylbutyl) benzene, 3,5,5-trimethylhexyl)-benzene, (3,5,5 - trimethylhexyl) toluene, (3,5,5-trimethylhexyl)-xylene, nonyltoluene (the nonyl group being from propylene trimer), decylbenzene, n-dodecylbenzene, dodecylbenzene or

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dodecyltoluene (the dodecyl group being from propylene tetramer), tetradecylbenzene, tetradecyltoluene, tetradecyldimethylbenzene, pentadecylbenzene, hexadecylbenzene, etc. These are halomethylated with a hydrogen halide and formaldehyde or paraformaldehyde or with the equivalent thereof conveniently in the presence of such a catalyst as zinc chloride in glacial acetic acid. There are thus obtained the chloromethyl or bromomethyl derivatives.

An alkylbenzyl halide and dimethylaminoacetone or diethylaminoacetone are mixed in about molar proportions and reacted together to give quaternary ammonium compounds. The reaction is desirably accomplished in the presence of a solvent, such as benzene, toluene, isopropyl alcohol, isopropyl ether, acetonitrile, formamide, nitromethane, or the like. Heating is useful in accelerating and/or completing the reaction, temperatures of 50° to 125° C. being generally suitable. The product is obtained as a residue upon evaporation of solvent, if used. The residue may be purified by stripping, extraction, or similar operation, if desired.

The preparations of typical quaternary ammonium salts of this invention is illustrated by the following examples, in which parts are by weight.

Example 1

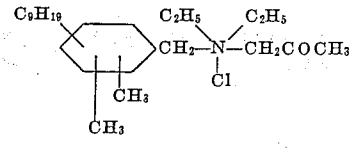
There were mixed 48 parts of octylbenzyl chloride, 20.2 parts of dimethylaminoacetone, and 120 parts of dry benzene. The mixture was heated under reflux for four hours, after which time it was found that the titratable nitrogen was essentially zero. The solvent was driven off to yield a viscous syrup. This was found to contain 3.9% of nitrogen and 10.1% of chlorine and to correspond in composition to octylbenzyl acetonyl dimethyl ammonium chloride, for which the theoretical nitrogen and chlorine contents are 4.1% and 10.4%, respectively.

This compound is readily soluble in water and is compatible with alkaline cleaning solutions. It has a phenol coefficient of 80 against *Staphylococcus aureus*.

Example 2

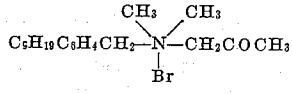
There were mixed 28 parts of a nonyldimethylbenzyl chloride, 13 parts of diethylaminoacetone, and 100 parts of toluene. The mixture was heated under reflux for five hours. The toluene was removed by heating of the mixture under reduced pressure to yield a gummy residue. This corresponds in composition to nonyldimethylbenzyl acetonyl diethyl ammonium chloride. It has a

phenol coefficient of 315 against *Staphylococcus aureus* and of 225 against *Salmonella typhosa*. This compound has the formula



Example 3

There were mixed 29.7 parts of nonylbenzyl bromide, 10.2 parts of dimethylaminoacetone, and 50 parts of acetonitrile, and the resulting mixture was stirred and heated for an hour at reflux temperatures and left standing overnight. The reaction mixture was heated under reduced pressure to remove solvent and extracted with petroleum ether. The residue was again stripped by heating under reduced pressure. The product thus obtained corresponds almost exactly to



This compound is readily soluble in water. It gives a phenol coefficient of 335 against *Staphylococcus aureus* and of 240 against *Salmonella typhosa*.

Example 4

A mixture of 17 parts of dimethylaminoacetone, 51 parts of 2-methyl-5-dodecylbenzyl chloride, and 120 parts of dry benzene was heated under reflux for three hours. A sample titrated for basic nitrogen showed that the reaction was essentially complete. Concentration under reduced pressure gave a quantitative yield of product in the form of a hygroscopic glass. Analysis of this material showed the presence of 8.6% of chlorine and 3.5% of nitrogen. It corresponds in composition to dodecylmethylbenzyl dimethyl acetonyl ammonium chloride, for which the theoretical chlorine content is 8.66% and the theoretical nitrogen content is 3.42%. It has a phenol coefficient of 375 against *Salmonella typhosa* and of 640 against *Staphylococcus aureus*.

Dodecylbenzyl dimethyl acetonyl ammonium chloride is prepared from dodecylbenzyl chloride and dimethylaminoacetone as above. It has properties very closely similar to those of the dodecylmethylbenzyl compound having phenol coefficients at least as high as those of the latter.

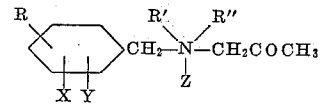
Example 5

By the procedure of Example 4 there are reacted 17 parts of dimethylaminoacetone and 57 parts of a pentadecylbenzyl chloride in which the pentadecyl group was derived from a propylene polymer cut with about 15 carbon atoms in the molecule. The product is pentadecylbenzyl dimethyl acetonyl ammonium chloride. It has a phenol coefficient of 600 against *Staphylococcus aureus* and 395 against *Salmonella typhosa*.

The alkylbenzyl acetonyl dialkyl ammonium halides of this invention have not only good bactericidal properties but also other significant and favorable characteristics. For example, they remain highly effective in hard water and in the presence of organic contaminants. This is a distinct advantage over many otherwise effective quaternaries. The compounds of this invention possess good solubility coupled with a favorable stability and it is of interest that the solubilizing group which is here present is not antagonistic to the action of the ammonium group as is often the case. The constituent parts and their particular configuration provide compounds which are advantageous with regard to properties and at the same time are advantageous because they are obtained from readily available and economical starting materials.

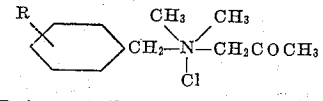
We claim:

1. As new chemical substances, compounds of the formula



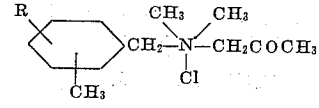
wherein R is an alkyl group of eight to sixteen carbon atoms, X and Y are members of the class consisting of hydrogen and the methyl group, R' and R'' are alkyl groups of not over two carbon atoms, and Z is a member of the class consisting of chlorine and bromine.

2. As new chemical substances, compounds of the formula



wherein R is an alkyl group of nine to sixteen carbon atoms.

3. As new chemical substances, compounds of the formula



wherein R is an alkyl group of nine to sixteen carbon atoms.

4. As a new chemical substance, nonylbenzyl dimethyl acetonyl ammonium chloride.

5. As a new chemical substance, nonylmethylbenzyl dimethyl acetonyl ammonium chloride.

6. As a new chemical substance, dodecylbenzyl dimethyl acetonyl ammonium chloride.

7. As a new chemical substance, dodecylmethylbenzyl dimethyl acetonyl ammonium chloride.

8. As a new chemical substance, pentadecylbenzyl dimethyl acetonyl ammonium chloride.

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REFERENCES CITED

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

Stevens: "J. Chem. Soc." (London), 1930, pp. 2119–2125.