

UNITED STATES PATENT OFFICE

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SALTS OF TERTIARY N-DI-ALKYL DERIVATIVES OF LONG CHAIN AMINES

No Drawing.

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This invention relates to new products suitable for use as wetting, cleansing and emulsifying agents.

In my co-pending application Serial No. 367,632 filed May 31, 1929, of which this is a continuation in part, I disclose the preparation of new compounds possessing qualities which make them very desirable as wetting, cleansing and emulsifying agents.

10 In general such materials are effective largely on account of their effect on the interfacial tension between water and an oily or greasy surface. The magnitude of this effect appears to depend on (a) the presence of certain groups in the molecule and (b) on the shape and general configuration of the molecule. By taking these factors into consideration I have discovered a series of substances which possess the above-named properties in a remarkable degree.

25 It is well known that ammonia and organic substituted ammonias such as the methylamines have cleansing properties. These almost entirely disappear however when these substances are converted into their salts, with, for example, the mineral acids. Thus a solution of ammonium chloride has practically no cleansing properties, and the same holds for methylamine hydrochloride. 30 Further up the homologous series n-amylamine is found to have considerable wetting properties while the corresponding hydrochloride is a very feeble wetting agent. In general the salts of such amines are far inferior in cleansing properties to the amines themselves. 35

I have now discovered a new series of compounds, the salts of tertiary alkyl amines, which, provided the alkyl groups are suitably selected, possess to a large degree the property of reducing the surface tension of aqueous solutions. To secure this effect I prepare a tertiary alkyl amine in which one alkyl radical consists of a long carbon chain 45 fully saturated with hydrogen, while the other two alkyl radicals have no more than four carbon atoms each. The degree of wetting which may be secured at low concentrations of these materials depends, other things being equal, on the length of the long

chain. By preparation of representative members of an extensive series I have found that with a long chain of seven carbon atoms the resulting solutions have feeble wetting properties, with a chain of eight carbon atoms the wetting properties are decidedly increased, with twelve they are still better and this improvement is maintained up to fifteen carbon atoms. 55

While the initial experiments were confined to straight chain compounds I have now discovered that branched chain compounds are also useful, provided the number of carbon atoms directly connected falls within the limits outlined above. For example, amine salts of this type derived from dicapryl alcohol are very useful. 65

For members at any one length of long chain, maximum reduction of surface tension is secured by having the two short chain radicals as small as possible, that is, methyl groups, but others are good. A further development in the case of the short chain radicals is the discovery that hydroxy derivatives of the alkyl radicals may also be used to good effect. 75

These materials may be prepared by heating the halogen derivative corresponding to the long chain alkyl radical with the N-di-n-alkyl amine selected; or the primary amine corresponding to the long chain alkyl radical may be alkylated by any of the standard methods. The salts are formed by neutralization with the acid chosen. The common mineral acids are most convenient, but the soluble organic acids such as acetic, oxalic, citric may also be utilized. 85

These salts are solids, generally of soap-like appearance. In some cases they may be prepared in crystalline form by recrystallization from organic solvents. They give aqueous solutions which foam readily and have wetting qualities at large dilutions. They are hence useful in the textile industry in washing, cleansing and wetting out being especially valuable in acid baths, and also in dispersing of certain dyestuffs. These solutions possess powerful emulsifying qualities for oils, and are valuable, for instance, in fatliquoring of leather. They have the prop- 90 95 100

erty of forming insoluble lakes with many acid or direct dyestuffs; this may be utilized in the mordanting of vegetable-tanned leather. Alternatively these materials may be used in assisting the penetration of basic dyes on vegetable tanned leather.

The following examples illustrate the method of preparation, but other standard methods may be used if desired. The invention is not however confined to these examples:

Example 1. N-di-n-butyllaurylamine bromide

25 grams lauryl bromide, 65 grams di-n-butylamine, are warmed up together under reflux condenser for 3 hours. Reaction occurs very readily and almost completely. Product is diluted with a large volume of water, and made acid. On ether extraction the bulk of the N-di-n-butyl lauryl amine salt goes into the ether, and on distilling off the ether a completely water soluble product is obtained.

Example 2. N-diethanollaurylamine sulphate

50 grams lauryl bromide, 110 grams diethanolamine, are warmed up together under reflux condenser for 7 hours, when a test drop from the solution gives a clear solution in acidified water. In this case the reaction proceeds practically to completion. The mixture is diluted with water, and extracted in alkaline state with ether. The ether layer is washed with water to remove the excess of diethanolamine, dried, and the ether distilled off. The free base is neutralized with the calculated amount of sulphuric acid. The salt thus obtained is completely soluble in water.

Example 3. N-diethyldicapryl oxalate

39 grams dicaprylbromide obtained from crude dicapryl alcohol, and boiling at 165 to 180° C. at 5 mm. pressure are mixed with 48 grams diethylamine and warmed up together under reflux condenser for 13 hours. The product is poured into alkaline water and extracted with ether. The ether extract is washed thoroughly, then acidified, and the acid solution made very dilute to take up nearly all the desired amine salt. After separation the aqueous layer is made basic, and extracted with ether. The ether extract is dried, the ether distilled off, and the product vacuum distilled. The portion boiling at 146.5 to 150° C. at 2 mm. pressure is collected. By neutralization in acetone solution with the calculated amount of oxalic acid and evaporation the pure salt is obtained.

It is understood that my invention is not confined to preparation of the compounds by the above methods nor to the materials mentioned since the examples are merely illustrative of the preparation of a representative number of members in the series.

I claim:

1. As new products the salts of amines of the general type



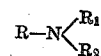
where R represents an alkyl radical containing from 9 to 15 carbon atoms directly connected in a straight chain, R₁ and R₂ represent radicals of not more than 4 carbon atoms of the class consisting of alkyl and hydroxy alkyl groups.

2. As new products the salts of amines of the general type



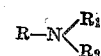
where R represents an alkyl radical containing from 9 to 15 carbon atoms directly connected in a straight chain, R₁ and R₂ represent alkyl radicals containing not more than 4 carbon atoms.

3. As new products the salts of amines of the general type



where R represents the dicapryl radical, R₁ and R₂ represent alkyl groups of not more than 2 carbon atoms.

4. As new products the salts of amines of the general type



where R represents the dicapryl radical, R₁ and R₂ represent ethyl groups.

5. As new products the salts of N-di-ethyl-dicapryl amine.

6. As a new product N-di-ethyldicapryl oxalate.

7. As a new product N-di-ethanollaurylamine sulphate.

In testimony whereof I affix my signature.
IAN C. SOMERVILLE.