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PROCESS OF PREPARING AMINO-SULPHONIC ACIDS SUBSTITUTED IN THE AMINO GROUP BY ACYL RADICALS OF FATTY ACIDS

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The present invention relates to a process of preparing amino-sulphonic acids substituted in the amino-group by acyl radicals of fatty acids.

We have found a process of preparing amino-sulphonic acids in the amino-group of which one hydrogen atom is substituted by the acyl radical of a fatty acid. The process consists in causing reaction products, obtained by the action of aldehydes or ketones on sulphites, to condense with fatty acid amides.

The fatty acid amides are easily accessible by dehydrating ammonium salts of fatty acids by a heat treatment or by dehydrating the fatty acids by a heat-treatment while passing ammonia gas through the fatty acids. The aldehyde bisulphites or ketone bisulphites are easily obtained in the usual manner known as determination method or purifying method.

In order to condense both products it suffices to mix them simply and to dehydrate them under atmospheric or reduced pressure by heating. No complicated apparatus, special vessels and troublesome processes are necessary for carrying out the process and, therefore, cheap technical production is possible.

The process may be carried out by heating an amide in the molten state, for instance, together with an aldehyde bisulphite or a ketone bisulphite, while well stirring. Mixtures from aldehyde bisulphite compounds and ketone bisulphite compounds may also be used.

In some cases it may be advantageous to work in the presence of an organic solvent of high boiling point, for instance xylene, dichlorobenzene or pyridine. When using one of the two first mentioned substances it is possible to remove the water of reaction, which has been formed, very easily from the mixture by means of azeotropic distillation. In many cases the said reactions may also be performed in an aqueous medium especially in case the carboxylic acid amide used as parent material is soluble in water or has a certain affinity for the water.

The temperature of reaction varies within wide limits according to the parent materials used. If amides of fatty acids of high molecular weight are used as parent material the most favorable temperature of reaction lies between 100° C. and 120° C. In general, the reactions may be carried out without application of pressure. Only in some cases it is necessary to work in closed vessels under pressure.

Since the carboxyl radical of the fatty acid in form of the acid amide is closed the products and their salts possess a high stability towards acids, bases and salts and very good properties as washing, wetting, emulsifying and softening agents. With regard to their washing power they correspond, if the conditions are the same, with all the products now in commerce.

As fatty acids which may be used in the process of the present invention there may be used above all the fatty acids having at least 6 carbon atoms, furthermore, there may be mentioned vegetable and animal fats, oils, waxes and resins or fatty acids contained in mineral oils or aromatic aliphatic acids.

The following examples serve to illustrate the invention but they are not intended to limit it thereto; the parts are by weight:

(1) Equal parts of oleic acid amide and of industrial sodium formaldehyde-bisulphite are heated together to 155° C., while stirring. After 1½ hours reaction set in with a slight rise of the temperature. The thinly-liquid mass, which at first froths feebly, becomes viscous in the course of a further 3 hours but can still be stirred. A test portion thereof dissolves even in cold water to a slightly turbid solution and has a very good foaming power. By stirring for further 9 hours at 155° C. a yellowish powder is obtained which dissolves in water to an entirely clear and colorless solution. The product which has been crystallized from dilute methanol is found by analysis to have the formula

$$C_{17}H_{33}CO.NH.CH_2.SO_3Na$$

Instead of the oleic acid amide the lauric acid amide may be used with the same success.

(2) 28 parts of oleic acid amide are dissolved in 30 parts of xylene. 32 parts of formaldehyde bisulphite of 80 per cent strength are added and the whole is heated in a still to 145° C. to 155° C., while stirring, the xylene which distils being replaced by the same quantity of xylene. In order to facilitate the distillation of the xylene and the water of reaction a slightly reduced pressure may be used. After stirring for several hours, the xylene is blown off by means of steam. The product dissolves in warm water to a clear solution while frothing strongly. The solution remains clear even on addition of dilute hydrochloric acid.

(3) 20 parts of commercial lauric acid amide and 45 parts of commercial acetaldehyde sodium bisulphite are heated, while stirring, for 4 hours at 155° C. After recrystallization, there is obtained a white powder which is soluble in water to a clear solution and foams very strongly even in an acid solution. For many commercial pur-

poses it is, however, not necessary to recrystallize the product, since it may also be used in the crude form.

(4) In the same manner as indicated in Example 3, a powder having the same good properties is obtained from 84 parts of commercial oleic acid amide and 138 parts of commercial acetone sodium bisulphite by heating the components for 3¼ hours at 155° C. and for 6¼ hours at 180° C.

(5) In the manufacture of soap fatty acids from paraffin there is also formed an important proportion of carboxylic acids containing 5 to 11 carbon atoms in the molecule which are as suitable for the present process as the soap fatty acids. An acid containing 7 carbon atoms is, by means of ammonia, transformed into the corresponding acid amide melting at 83° C. to 89° C. A mixture of 39 parts of this acid amide and 84 parts of commercial formaldehyde sodium bisulphite is maintained, for 3 hours, at a temperature of 155° C. Stirring is continued for some time and there is thus obtained, without any further purification, a product which dissolves in water to a clear solution and gives rise to the formation of a strong foam.

(6) 280 parts of oleic acid amide obtained from technical oleic acid are mixed with 220 parts of formaldehyde-bisulphite paste of 90 per cent strength, then melted by heating and stirred in order to evaporate water contained therein. The temperature is raised to 120° C. to 170° C. and then the product is allowed to stand at this temperature until a test portion taken from it dissolves in water to a clear solution. A white condensation product, forming a powder, is obtained which possesses a high wetting and washing effect and shows strong stability towards acids, bases and salts.

(7) To a mixture consisting of 90 parts of oleic acid amide and 10 parts of oleic acid 80 parts of a formaldehyde-bisulphite paste of 90 per cent. strength are added and the whole is heated for 5 to 6 hours at 120° C. to 160° C. in order to remove water contained therein. A product is obtained, forming a powder which has the same good properties as the product obtained according to the process of Example 6. It is distinguished from the product of Example 6 by a higher solubility in cold water and much better stability towards acids and hard water.

(8) 100 parts of sperm oil acid amide are mixed with 250 parts of a formaldehyde-bisulphite paste of 90 per cent. strength and the whole is then worked up as described in Example 6. A product is obtained which possesses a very good washing powder and is distinguished especially as emulsifying agent.

(9) 510 parts of a solution of 50 per cent strength of neutral sodium sulphite are mixed with 175 parts of aqueous formalin of 35 per cent. strength. The alkali obtained as a by-product is neutralized by means of sulphuric acid, water is evaporated and 200 parts of the formaldehyde-bisulphite paste of 80 per cent. strength obtained are mixed with 150 parts of coconut oil acid amide. By carrying out the reaction at 160° C. in the same manner as described in Example 6 a white product is obtained which is dissolved in the quantity of sulphuric acid necessary to decompose the formaldehyde-bisulphite in excess and then again neutralized by means of caustic alkali. A white paste-like washing and wetting agent is obtained which is very stable towards hard water and acids.

(10) 1 part of abietic acid amide is intimately triturated together with 1 part of formaldehyde-sodium-bisulphite and the mixture is heated in an oil bath while stirring, for 3¼ hours at 150° C. to 160° C. and for further 4½ hours at 180° C. to 185° C. After entire removal of unaltered abietic acid amide by means of acetone there are obtained 1.4 parts of a reaction product which dissolves in water to a clear, foaming solution. The sodium abietinoylaminomethane-sulphonate is a light yellow powder.

We claim:

1. The process of producing a compound having the following formula:

$$C_{17}H_{33}CO.NH.CH_2.SO_3H$$

and its sodium salt, which process comprises condensing a mixture of oleic acid and a member selected from the group consisting of sodium formaldehyde bisulphite and formaldehyde bisulphite by heating the mixture to a temperature which evaporates the water formed by the condensation of the reagents and continuing the heating until the condensation is complete, the reaction being carried out at pressures not exceeding atmospheric and in the absence of diluents and catalysts.

2. The process of manufacturing amino sulphonic acids substituted in the amino group by an acyl radical of a fatty acid containing at least six carbon atoms, which comprises condensing an amide of a fatty acid containing at least six carbon atoms with the reaction products resulting from bisulphites and a member of the group consisting of aldehydes and ketones, the condensation being carried out by heating to a temperature which causes evaporation of the water formed during the condensation and at a pressure not exceeding atmospheric, said process being carried out in the absence of diluents and catalysts.

3. The process as in claim 2 in which the amide is condensed with a formaldehyde bisulphite compound.

4. A process as in claim 1 in which the temperature at which the condensation is effected is 155° C.

5. A process as in claim 2 in which lauric acid amide is condensed with acetaldehyde sodium bisulphite.

6. A process as in claim 2 in which oleic acid amide is condensed with acetone sodium bisulphite.

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