

1

2,816,920

PRODUCTION OF ZWITTERION OF DETERGENT AMINO ACIDS

Donald L. Andersen, Minneapolis, Minn., assignor to General Mills, Inc., a corporation of Delaware

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The present invention relates to the production of the zwitterion from the salts of certain detergent amino acids and more particularly to the production of the zwitterion substantially free from inorganic salt contamination.

The salts of the detergent amino acids to which the present invention is directed are those having the following formula:



in which R is an aliphatic hydrocarbon group containing from 8–22 carbon atoms and is derived from a detergent forming fatty acid, R' is H or CH₃, Me is alkali metal, m is 0 or 1, n is 1 or 2 but 1 when R' is CH₃ and the sum of m and n is 2.

Detergent materials of the above formula are frequently made in the form of the sodium or potassium salts and are frequently the hydrolysis products of the corresponding esters or nitriles. The sodium salt of compounds of this type is generally quite alkaline in reaction. For example, sodium dodecylaminopropionate in a 10% aqueous solution has a pH in excess of 12. This is too alkaline for uses in which the material comes in contact with human skin as, for example, in the case of shampoos. Neutralization with mineral acid to an acceptable pH level produces considerable inorganic salt. This inorganic salt frequently cannot be tolerated because of solubility and viscosity considerations.

For these reasons it is frequently desirable to convert the sodium or potassium salt to some other more desirable salt and for this purpose the free acid form of the compound is a desirable starting material.

When an excess of mineral acid is added to solutions of the sodium salts of the above detergent amino acids, the zwitterion can be precipitated but in a semi-solid form which is extremely difficult to handle and to purify. Considerable inorganic salt and mineral acid are occluded within the semi-solid mass. The zwitterion thus produced is difficult to obtain in a salt-free form because the physical form makes washing very tedious.

It has now been discovered that the zwitterion of the detergent amino acids can readily be obtained and purified by a unique procedure.

It is, therefore, an object of the present invention to provide a novel process of producing the zwitterion of the above detergent amino acids and of purifying the zwitterion by a simple series of operations.

It has been discovered that by a careful neutralization of a 10–30% aqueous solution of an alkali metal salt of a detergent amino acid of the above type, by means of sulfuric acid to a pH within the range of 3.5 to 5.5, there results a rapid and complete separation of the reaction mixture into 2 phases. The upper phase comprises a 20–50% aqueous solution containing virtually all of the detergent material contaminated to a slight degree with sodium sulfate dissolved in the solution. This phase is a slightly viscous, clear and readily handled solution. The lower aqueous phase is a solution of sodium sulfate containing only a trace of the de-

2

tergent amino acid. The separation is rapid and complete with no emulsion problems and yields approximately one part of organic phase to 2–3 parts of aqueous phase.

This organic phase may be used directly for the production of other salts of the amino acid where a slight amount of sodium sulfate or other inorganic salt may be tolerated. It is preferred, however, to isolate the zwitterion in substantially pure form. This may readily be accomplished by adding the organic phase slowly to a quantity of water about 2–3 times that of the organic phase, while the water is being well agitated. The zwitterion is precipitated as a finely divided flocculent semi-solid. The particles of this flocculent dispersion remain suspended as long as agitation is continued. When agitation is discontinued, the particles float to the surface as a fairly compact mass. The excess water may be decanted and the compact mass may be resuspended in added wash water by simple agitation. Washing and decanting may be repeated any number of times to yield a product which is substantially free from inorganic salts, without any significant loss of the zwitterion. A total of 3 such washings is usually sufficient to leech out virtually all of the inorganic salt. Upon completion of the washing, the zwitterion may be separated by decantation after which residual amounts of free water may be removed by kneading or pressing. As an alternative, the residual water may be removed by melting the zwitterion and then decanting any free water. The resultant product in either event is a gelatinous solid containing approximately 35% water, and is insoluble in water but soluble in alkaline materials.

While sulfuric acid is the preferred acid in that it is less expensive, less corrosive, and requires less accurate control, other known non-oxidizing mineral acids such as hydrochloric and phosphoric may also be used with essentially the same results.

Example 1

A solution of 20.9 lbs. of sodium β -dodecylaminopropionate in 88 lbs. of water was prepared in a 20 gallon autoclave equipped with an anchor stirrer. To this solution was added slowly and with agitation a solution of 5.2 lbs. of concentrated sulfuric acid in 15 lbs. of water to yield a mixture having a pH of approximately 4.0. Phase separation occurred almost immediately and, upon discontinuing the agitation, the organic phase floated to the top of the aqueous phase and the latter was separated by decantation, sampled for analysis and discarded. The upper organic phase weighed 50 lbs. and was washed by pouring it in the form of a thin stream into 15 gallons of water under agitation. The organic phase was again permitted to float and was separated by decantation. The organic phase was subjected to 2 additional washings with 10 gallons of water each. The wash waters were sampled for analysis and then discarded. The final product was approximately 65% solids and had an ash content of less than 0.5%. The following table gives the analyses of the original aqueous phase and the various wash waters.

Solution	Percent solids	Percent ash	Percent loss organic
Aqueous phase.....	6.14	6.11	0.1
1st wash.....	0.73	0.72	0.05
2d wash.....	0.25	0.23	0.07
3d wash.....	0.09	0.06	0.07

Example 2

The process of Example 1 was repeated with β,β' -dodecylaminobispropionate. The zwitterion was obtained in

3

essentially the same yield and purity for the zwitterion reported in Example 1.

Example 3

The process of Example 1 was repeated on sodium β -dodecylaminocrotonate with essentially the same results as reported in Example 1.

Example 4

The process of Example 1 was carried out substituting an equivalent quantity of hydrochloric acid for the sulfuric acid. The results obtained were essentially the same.

I claim as my invention:

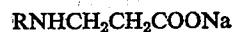
1. Process of preparing a zwitterion of a compound having the formula $\text{RNH}_m(\text{CHR}'\text{CH}_2\text{COOMe})_n$ in which R is an aliphatic hydrocarbon group containing from 8-22 carbon atoms, R' is selected from the group consisting of hydrogen and methyl, m is not greater than 1 and n is at least 1 and not more than 2 but is 1 when R' is CH_3 and the sum of m and n is equal to 2 and Me is alkali metal which comprises adding a nonoxidizing mineral acid to an aqueous solution of said alkali metal salt to produce a pH within the range of 3.5 to 5.5 while agitating the solution, discontinuing the agitation to permit the precipitated zwitterion thus formed to separate as an aqueous solution in the form of a distinct phase and separating the zwitterion phase.

4

2. A process according to claim 1 in which the mineral acid is sulfuric acid.

3. A process according to claim 1 in which the separated zwitterion is further purified by dispersing the zwitterion in water with agitation, discontinuing the agitation and permitting the zwitterion to accumulate as a distinct phase.

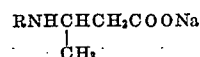
4. Process according to claim 1 in which the compound has the formula:



5. Process according to claim 1 in which the compound has the formula:



6. Process according to claim 1 in which the compound has the formula:



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