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INACTIVATION OF PRIMARY AMINES IN β-ALANINE DETERGENT MIXTURE COMPOSITIONS

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The present invention relates to the inactivation of primary amines in β-alanine detergent mixture compositions. The β-alanine detergents contemplated by the present invention are those having the following formula: $RNHCH_2CH_2COOH$ in which R is the long-chain aliphatic hydrocarbon group containing from 8 to 22 carbon atoms. In place of the acid the salts and esters may be used.

These detergents may be made as disclosed in Isbell Patent 2,468,012. According to that patent primary fatty amines are condensed with methyl acrylate to obtain the ester addition compound. This compound may then be hydrolized to the acid or converted to a salt such as the alkali metal salts, ammonium salts, and other organic base salts.

Heretofore it has not been considered that any free primary amine remains in the reaction mixture. It has now been found however, that under some reaction conditions a small quantity of free primary amine may remain in the reaction mixture and is difficult to remove. These free primary amines may cause skin disorders and accordingly it is desirable to obtain a detergent composition which is free from the primary fatty amines. It has now been discovered that by reacting the detergent composition with an aldehyde that the fatty primary amine may be modified in such a way that the detergent composition does not cause skin disorders in case of persons sensitive to primary amines.

It is therefore an object of the present invention to provide a process of inactivating primary amine in β-alanine detergent mixture compositions by means of an aldehyde. It is also an object of the present invention to provide a novel β-alanine detergent mixture composition in which free primary fatty amine has been inactivated by means of an aldehyde.

As disclosed in the Isbell patent a wide variety of primary fatty amines may be employed for the preparation of these detergents. These amines may contain from 8 to 22 carbon atoms and may be the mixed amines obtained from the mixed fatty acids of an oil or from any selected individual acid or any selected group of acids. The amines may be the saturated or unsaturated. These amines may be reacted with an acrylate ester especially the lower aliphatic acrylate esters in which the alcohol group of the ester contains from 1 to 4 carbon atoms.

For the purpose of inactivating free primary amines it is preferred to treat the ester addition compound thus obtained rather than any of the hydrolysis products such as the acid or salts. However, it is also possible to inactivate the primary amine by treatment either of the acid obtained by hydrolysis of the ester or by treating the alkali metal salts such as sodium and potassium salts of the acid.

The process of inactivating the primary amines involves the simple addition of an aldehyde to the detergent product. A wide variety of aldehydes may be employed including both aromatic and aliphatic aldehydes. Benzaldehyde and salicylaldehyde has been found to be particularly effective. Likewise the aliphatic aldehydes such as butyraldehyde has been found to be very effective. Other aldehydes such as formaldehyde and glyoxal may also be used but are generally not as desirable as some of the preferred aldehydes referred to above.

The aldehyde is merely added to the reaction mixture and no special reaction conditions are required. The reaction proceeds at room temperature in a reasonable period of time. Thus since these detergents are not ordinarily used immediately the aldehyde may simply be added to the reaction mixture and reaction will have been completed by the time that the detergent is used after having distributed through the normal channels of commerce. If desired the reaction may be speeded up by employing higher temperatures. Likewise it is not necessary to employ solvents, catalysts or other reaction aids, although, of course these may be used if desired. It is preferred to employ approximately an amount of the aldehyde equivalent to the free primary fatty amine. Generally this will result in a reduction of the free amine content to considerably less than 1% even when starting with a detergent composition containing as high as 10% of the free primary fatty amines. Naturally, if desired, excess aldehyde may be used.

Example 1

To prepare the β-alanine methyl ester, distilled amine derived from coconut fatty acids was used. This product had an amine number of 274.6. Amine number is defined as the mg. of KOH equivalent to the free amine in 1 g. of product. It is determined by titrating a solution of the amine in ethyl alcohol toluene to a bromcresol green end point with aqueous hydrochloric acid. Also, it may be determined by titrating with perchloric acid in anhydrous acetic acid solution, as described by Terry, Eilar, and Moe in Analytical Chemistry (volume 24 for 1952).

The coco amine (306 g.) was heated to 100° C. under a vacuum of 10 mm. Thereafter, it was cooled to 40° C. and methyl acrylate (142 g.) was added over a period of 8 minutes. The mixture was agitated at 40° C. for 6 hours. Vacuum was applied and the mixture was heated to 100° C. at 40μ to remove volatile material. The product weighed 438 g. It had an amine number of 188.6, a primary amine content of 8.2 and a secondary amine content of 83.9. To this ester (20 g.) was added salicylaldehyde (1.1 g.) and the solution was allowed to stand for 24 hours. The primary amine content at the end of this time had decreased to 0.5%.

Example 2

The above experiment was repeated save that 0.96 g. of benzaldehyde was used instead of salicylaldehyde. In this instance the final product had a primary amine content of 0.

Example 3

A β-alanine ester was prepared as described in Example 1 using 1,875 g. of a distilled coco amine with an amine number of 269.6 and 1,170 g. of methyl acrylate. Here a reaction temperature of 90° C. was used with agitation for 4 hours. The yield of product was 2,735 g. The ester had the following constants:

Amine number	170.9
Primary amine content	1.4
Secondary amine content	81.2
Tertiary amine content	13.7

This product (357 g.) was mixed with butyraldehyde (12 g.) and allowed to stand overnight at room tem-

perature. The primary amine content of the treated material decreased from 1.4% to 0.09%.

We claim as our invention:

1. A process of inactivating free primary amines in a β-alanine detergent composition in which the β-alanine contains an aliphatic hydrocarbon substituent of from 8 to 22 carbon atoms on the amino nitrogen, which comprises reacting said β-alanine detergent composition with an aldehyde which is reactive with primary amines to produce products inert to human skin.

2. A process of inactivating free primary amines in a β-alanine detergent composition in which the β-alanine contains an aliphatic hydrocarbon substituent of from 8 to 22 carbon atoms on the amino nitrogen which comprises reacting said β-alanine detergent composition

with a quantity of an aldehyde substantially equivalent to the free primary amine content of the detergent composition, said aldehyde being reactive with primary amines to produce products inert to human skin.

3. Process according to claim 1 in which the aldehyde is benzaldehyde.

4. Process according to claim 1 in which the aldehyde is salicylaldehyde.

5. Process according to claim 1 in which the aldehyde is butyraldehyde.

References Cited in the file of this patent

UNITED STATES PATENTS

2,468,012 Isbell ---------- Apr. 19, 1949