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## PROCESS OF PREPARING AMINE SULFONATES AND PRODUCTS OBTAINED THEREOF

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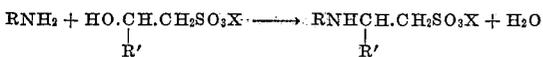
7 Claims. (Cl. 260—513)

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The present invention relates to methods of preparing taurine derivatives and provides an improved method for the manufacture of higher N-alkyltaurines and valuable surface-active agents comprising certain new taurine derivatives.

An object of the invention is the provision of a commercially feasible method for the preparation of salts of N-alkyltaurines in which the alkyl radical has from 8 to 18 carbon atoms. Another object of the invention is to prepare compounds possessing very good surface-active properties from readily available raw materials. Still another object of the invention is to provide compounds which simultaneously possess superior 15  
detergent, wetting-out and lathering properties.

These and other objects hereinafter disclosed are provided by the following invention wherein an isethionic acid compound selected from the class consisting of isethionic acid and 2-methylisethionic acid and the alkali metal, alkaline earth metal, and ammonium salts of such acid is heated with an alkylamine of from 8 to 18 carbon atoms to yield N-alkyltaurines or the corresponding salts thereof in which the alkyl radical has from 8 to 18 carbon atoms, substantially according to the scheme:



in which R is an alkyl radical of from 8 to 18 carbon atoms, R' is selected from the class consisting of hydrogen and the methyl radical and X is selected from the class consisting of hydrogen, alkali metal, alkaline earth metal and ammonium.

Alkylamines suitable for the present purpose are, for example n-octylamine, (2-ethylhexyl)amine, n-decylamine, tert-dodecylamine, (2-butyl-octyl)amine, n-undecylamine, n-tetradecylamine, (7-ethyl-2-methyl)undecylamine, n-hexadecylamine, n-heptadecylamine, n-octadecylamine, etc. Isethionic acid compounds which may be condensed with the alkylamines for the preparation of the present N-alkyltaurines are, e. g., isethionic acid or 2-methylisethionic acid, sodium isethionate, sodium 2-methylisethionate, potassium isethionate, lithium isethionate, magnesium 2-methylisethionate, calcium isethionate, barium 2-methylisethionate, ammonium isethionate, etc. The isethionates or the 2-methylisethionates are readily available by the reaction of an alkali metal or alkaline earth metal bisulfite with ethylene oxide or propylene oxide.

Processes previously employed for the prepara-

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tion of higher N-alkyltaurines have been inexpedient for commercial operation because they either required the use of difficultly available starting materials or because yields of the desired materials, based on the quantity of amino compound used, were very low due to the formation of large quantities of by-products. Thus in the German Patent 551,257 issued to Ott et al., there is described the production of the sodium salt of N-heptadecyltaurine by condensation of heptadecylamine with sodium 2-chloroethanesulfonate. In this process, a part of the heptadecylamine is neutralized by the hydrogen chloride evolved in the condensation; hence the process is of little commercial value in that not only is a considerable excess of initial amine required, but also there result operating difficulties in the separation by by-product heptadecylamine hydrochloride from the N-heptadecyltaurine.

In prior art, e. g., as in the Nicodemus U. S. Patent No. 1,932,907, N-alkylamines in which the alkyl radical has from 1 to 4 carbon atoms have been condensed with isethionic acid or salts thereof in aqueous solution and under superatmospheric pressure to yield the corresponding lower N-alkyltaurines, i. e., taurines in which the alkyl radical has from 1 to 4 carbon atoms. Now I have found that in spite of the fact that superatmospheric pressure was employed when working with the comparatively more reactive lower alkylamines, condensation of the usually more sluggish higher alkylamines with isethionic acid or salts thereof may be readily effected under ordinary conditions of pressure.

In preparing the higher N-alkyltaurines or salts thereof by the present process, I operate substantially as follows: I heat a mixture of an alkylamine of from 8 to 18 carbon atoms and the isethionic or 2-methylisethionic compound thereof to temperatures of from, say, 180° C. to 300° C. until the formation of the higher N-alkyltaurine compound. Advantageously, I employ no water or other diluent, although anhydrous conditions need not be observed. Since the reaction occurs by condensation of one mole of the alkylamine with one mole of the hydroxyalkylsulfonate, equimolar proportions of the reactants generally may be employed. In practice, it may be preferable to use a slight excess of whichever component is more readily obtainable in order to assure complete reaction of the less readily obtainable reactant. The reaction product, i. e., the higher N-alkyltaurine compound is separated from the reaction mixture by simply removing any unreacted constituent, e. g., by distilling, decanting or

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extracting, and drying the product. Generally, the use of substantially equimolar proportions of the alkylamine and the isethionic compound and heating at temperatures of over 180° C. for a time of, say, from one hour to a day, assures complete reaction of both components so that the reaction product consists of the substantially pure, waxy or crystalline higher N-alkylamine compound. In this case no further treatment of the product is required.

Higher N-alkyltaurines prepared by the present process are white, waxy to crystalline solids. The alkali metal and ammonium salts are generally water-soluble and, like most long-chained sulfonates, the individual members of this series are generally characterized by possessing varying degrees of surface-activity. While some members of the series are distinguished by good wetting properties, very good deterative properties or outstanding lathering ability, generally a single member of the series does not possess all three of these desirable characteristics, e. g., outstanding deterative properties is present in a compound having poor lathering properties, or the good wetting-out property of a single member is not accompanied by adequate deterative and/or lathering properties. I have found, however, that salts of N-tetradecyltaurine, and particularly the alkali metal and ammonium salts thereof, are unique in that they are characterized by possessing a combination of outstanding deterative, wetting-out, and lathering properties. The provision of a single compound possessing such general superiority constitutes a notable advancement in the art.

The invention is illustrated, but not limited, by the following examples.

#### Example 1

A mixture consisting of 50 g. (0.338 mole) of sodium isethionate and 80 g. (0.362 mole) of N-tetradecylamine was heated rapidly to a temperature of 20° C. and maintained for 5 minutes at this temperature. The temperature of the reaction mixture was then slowly raised to 245° C. and maintained at about this temperature, with stirring, for about 2 hours. After allowing the resulting reaction mixture to cool it was extracted with 250 ml. of ethanol in order to remove excess amine. Upon drying there was obtained 95.1 g. (80.5% theoretical yield) of the sodium salt of N-tetradecyltaurine. As shown in Example 6 it was found to possess outstanding deterative, wetting-out and lathering properties.

#### Example 2

50 g. (0.388 mole) of sodium isethionate was mixed with 75 g. (0.565 mole) of N-octylamine in an atmosphere of nitrogen at a temperature of about 150° C. and the resulting mixture was maintained, with stirring, at about the melting point of the sodium isethionate (about 181° C.) for several hours. Unreacted amine was then removed from the cooled product by extracting with ethanol. The dried residue comprised the substantially pure sodium salt of N-octyltaurine.

#### Example 3

50 g. (0.388 mole) of sodium isethionate was mixed with 65 g. (0.350 mole) of N-dodecylamine in an atmosphere of nitrogen at a temperature of from 225-230° C. for 3 hours and then at a temperature of 250° C. for 1.5 hours. The white, waxy, solid obtained upon cooling the reaction mixture in an atmosphere of nitrogen

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comprised the crude sodium salt of N-dodecyltaurine. Removal of unreacted dodecylamine from the crude product was effected by heating it with absolute ethanol and separating the resulting ethanol solution of unreacted amine. The residue comprised the substantially pure sodium salt of N-dodecyltaurine which was found to show very good deterative properties.

#### Example 4

In this example there is employed an alkylamine prepared from the mixture of alcohols obtained by hydrogenation of coconut-oil fatty acids, which amine will be hereinafter referred to as cocoamine. 70 g. (0.340 mole) of the amine was mixed with 50 g. (0.338 mole) of sodium isethionate in a nitrogen atmosphere, the mixture was heated rapidly to a temperature of 205° C. kept at this temperature for 30 minutes and then slowly raised to 225° C. The mixture was then maintained, with stirring, at a temperature of from 220-250° C. for a time of about 2 hours. In the course of the heating 4.3 g. of the amine distilled over with the water of the reaction. The distilled amine, together with 10 more grams of cocoamine, was returned to the reaction mixture. After cooling the reaction mixture to room temperature in an atmosphere of nitrogen it was removed from the reaction vessel and the unreacted amine was extracted with slightly alkaline ethanol. There was thus obtained 89.5 g. of dried residue comprising the substantially pure sodium salt of N-alkyltaurine in which the alkyl radical is derived from coconut oil fatty acids.

#### Example 5

85.0 g. (0.316 mole) of octadecylamine and 44.5 (0.30 mole) of sodium isethionate were mixed and heated in an atmosphere of nitrogen at 225-230° C. for 4.5 hours and then at 245° C. for 1.5 hours. After cooling, the reaction product was dissolved in aqueous alcohol. Separation and drying of the aqueous layer gave the sodium salt of N-octadecyltaurine, a substantially water-insoluble compound which showed no surface activity.

#### Example 6

This example shows deterative, wetting-out, and lathering efficiencies of the sodium salt of N-tetradecyltaurine prepared in Example 1.

Deterative efficiency was determined by employing the method described by J. C. Harris in Soap and Sanitary Chemicals, for August 1943, in which method the detergency is expressed as compared to that of Gardinol WA, a commercial detergent produced by sulfating the alcohols derived by hydrogenation of coconut oil fatty acids. The speed of wetting was measured by the Draves Test. The foaming properties were measured by the Ross-Miles lather test, a proposed method of the American Society for Testing Materials. The following results were obtained.

65	Detergency, percent of Gardinol:	
	100% active—	
	In 50 p. p. m. water hardness ----	125
	In 300 p. p. m. water hardness ----	100
	Built—	
	In 50 p. p. m. water hardness ----	114
	In 300 p. p. m. water hardness ----	106

<sup>1</sup>40% of the sodium salt of N-tetradecyltaurine and 60% of builder consisting of 96.2% sodium sulfate, 1.53% of diethylene glycol, and 1.75% of anhydrous sodium phosphate.

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Speed of wetting in seconds (Draves):

At 0.5% concentration .....	18.8
At 0.25% concentration .....	18.3
At 0.125% concentration .....	33.0
At 0.0625% concentration .....	129.0

Ross-Miles lather heights (cm.):

In 50 p. p. m. water hardness—	
At once .....	12.9
After 5 min. ....	12.9
In 300 p. p. m. water hardness—	
At once .....	16.1
After 5 min. ....	16.0

What I claim is:

1. The process of producing N-alkyltaurines having the formula  $RNHC(R')H_2CH_2SO_3X$  in which R is an alkyl radical of from 8 to 18 carbon atoms, R' is selected from the class consisting of hydrogen and the methyl radical and X is selected from the class consisting of hydrogen, alkali metal, alkaline earth metal and ammonium which comprises heating, at atmospheric pressure and at a temperature of 180° C.-300° C., an alkylamine of from 8 to 18 carbon atoms with a compound selected from the class consisting of isethionic acid and 2-methylisethionic acid and the alkali metal, alkaline earth metal and ammonium salts of said acids.

2. The process of producing alkali metal salts of N-alkyltaurine in which the alkyl radical has from 8 to 18 carbon atoms which comprises heating, at atmospheric pressure and at a temperature of 180° C.-300° C., an alkali metal isethionate with an alkylamine of from 8 to 18 carbon atoms.

3. The process of preparing the sodium salt of

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N-tetradecyltaurine which comprises heating tetradecylamine with sodium isethionate at atmospheric pressure and at a temperature of 180° C.-300° C.

4. The process of preparing the sodium salt of N-octyltaurine which comprises heating octylamine with sodium isethionate at atmospheric pressure and at a temperature of from 180° C.-300° C.

5. The process of preparing the sodium salt of N-dodecyltaurine which comprises heating dodecylamine at atmospheric pressure and at a temperature of from 180° C.-300° C.

6. The process of preparing the sodium salt of an N-alkyltaurine in which the alkyl radical is derived by hydrogenation of coconut oil fatty acids which comprises heating, at atmospheric pressure and at a temperature of 180° C.-300° C., sodium isethionate with an alkylamine in which the alkyl radical is derived by hydrogenation of coconut oil fatty acids.

7. The process of preparing the sodium salt of N-octadecyltaurine which comprises heating octadecylamine with sodium isethionate at a temperature of from 180° C.-300° C. and at atmospheric pressure.

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References Cited in the file of this patent

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