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AMINE SALTS OF THE TRIDECYL ESTERS OF SULFOBENZOIC ACID

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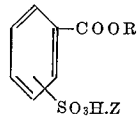
12 Claims. (Cl. 260—470)

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The present invention relates to surface-active agents and is particularly concerned with alkylamine or alkanolamine salts of tridecyl sulfobenzoates as new compounds possessing a high degree of utility as wetting-out and lathering agents.

As is known, surface-active agents find wide application in a large number of industrial processes, especially in those relating to the treatment of textiles and other fibrous materials. Among the compounds heretofore suggested for these and related purposes are the esters of sulfobenzoic acid and aliphatic alcohols containing from 8–12 or 18 carbon atoms, for example, the octyl, dodecyl, or oleyl sodium sulfobenzoate as illustrated in U. S. Patents 2,359,291 and 2,423,972. I have now found, as a result of comparative tests, that the heretofore unknown alkylamine or alkanolamine salts of the esters of sulfobenzoic acids and a certain mixture of isomeric tridecyl alcohols are of outstanding value, not only for the purpose of assisting in the wetting of textile materials, but also from a lathering standpoint. They are particularly valuable as tasteless foaming agents in dentifrice formulations, in edible oils adapted for use in mayonnaise preparation, and as tasteless wetting agents in the manufacture of food extracts, e. g., in soluble coffee extraction, etc. The present sulfobenzoates are obtainable by neutralizing with an alkylamine or an alkanolamine, the acid ester obtained by condensing 2-, 3- or 4-sulfobenzoic acid or an acid halide thereof with an alcohol fraction derived by the carbonylation and hydrogenation (Oxo) reaction of the olefins triisobutylene and tetrapropylene. The alcohol fraction which forms the starting material for my present esters may be defined as having the structure ROH, wherein R is a primary alkyl radical having an average of 13 carbon atoms derived from an alcohol formed by the carbonylation and hydrogenation of triisobutylene or tetrapropylene or mixtures thereof.

The present sulfobenzoates may be represented by the following structure



in which R is as defined above and in which Z is selected from the class consisting of mono-, di- and trialkylamines in which the alkyl radical contains from 1 to 12 carbon atoms, mono-, di- and trialkanolamines in which the alkanol radical

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has from 1 to 4 carbon atoms and alkylenepolyamines and polyalkylenepolyamines having from 2 to 4 amino groups in the molecule and from 2 to 3 carbon atoms in the alkylene chain. As examples of salts of tridecyl 2-, 3- or 4-sulfobenzoate having the above formula and provided by the present invention may be mentioned monoalkylamine salts such as the ethylamine, n-butylamine, 2-ethylhexylamine, n-decylamine and tert-dodecylamine salts; the dialkylamine salts such as the dimethylamine, diisopropylamine, di-n-octylamine and the methylethylamine salts; the trialkylamine salts such as the trimethylamine, tri-n-butylamine, and tri-tert-amylamine salts; the mono-alkanolamine salts such as the ethanolamine and the 4-hydroxybutylamine salts, the di-alkanolamine salts, such as the diisopropanolamine, dimethanolamine, and methanolethanolamine; the tri-alkanolamine salts such as the triethanolamine and tri-n-propanolamine salts; the alkylene or polyalkylene polyamine salts such as the ethylenediamine, triethylenetetramine, dipropylenetriamine, and diethylenetriamine salts, and morpholine.

The invention is further illustrated, but not limited, by the following example:

EXAMPLE

Into a reaction vessel equipped with mechanical stirrer, thermometer, and drying tube there was placed 310.5 g. (1.5 mole) of an isomeric mixture of tridecyl alcohols boiling within the range of 252.8° C.–273.9° C. and obtained by the Oxo process from a polymeric olefin of 12 carbon atoms, carbon monoxide and hydrogen. m-Sulfobenzoic acid (303.0 g.) was heated to 150° C. and then poured into the tridecyl alcohol, whereupon the temperature of the reaction mixture rose to 90° C. The resulting mixture was stirred at a temperature of from 90–95° C. for 80 minutes and then allowed to cool. At this point the reaction mixture comprised the substantially pure tridecyl sulfobenzoic acid. It was divided into 100 g. portions and each portion was then neutralized to the pH shown below with an ethanol solution of one of the following amines:

I monoethanolamine to pH 8.1
II triethanolamine to pH 6.55
III N-butylamine to pH 8.5
IV diisobutylamine to pH 7.1
V 2-ethylhexylamine to pH 7.25
VI diethylamine to pH 6.9
VII diethylenetriamine to pH 7.8

The resulting neutralized ethanol solutions were dried with stirring under reduced pressure (15–20

mm.) at a maximum pot temperature of 40–45° C. The dried products comprised the respective amine salts of tridecyl sulfobenzoate. Testing of the surface activity of the dried salts thus obtained gave the following values:

Surface activity of tridecyl sulfobenzoate amine salts

Amine Salt	Surface Tension dynes/cm., at percent Concentration			Interfacial Tension dynes/cm., at percent Concentration		
	1.0	0.25	0.0625	1.0	0.25	0.0625
monoethanolamine	29.7	30.7	31.3	2.4	4.9	7.4
triethanolamine	31.6	27.8	35.5	4.5	7.5	10.3
n-butylamine [1]	32.7	32.4	32.4	1.0	0.6	0.8
di-isobutylamine [1]	37.4	33.7	39.7	4.6	6.0	10.3
2-ethylhexylamine [1]	32.0	38.2	36.6	1.9	5.3	8.4
diethylamine	34.3	37.6	40.8	2.3	3.5	4.6
diethylenetriamine [1]	31.3	32.4	32.4	1.0	1.3	13.1

[1] For surface tension determination there were employed solutions in a liquid paraffinic hydrocarbon known to the trade as Nujol and having a surface tension of 38 dynes/cm. Interfacial tension was determined against distilled water.

Evaluation of the water soluble salts prepared above for wetting-out efficiency was conducted by employing the Draves Test of the American Association of Textile Chemists. The wetting speeds thus obtained are shown in the following table:

Salt Tested	0.5	0.25	0.125	0.0625	0.03	0.01
monoethanolamine	3.7	6.2	14.2	36.8	75.0	180+
triethanolamine	3.9	6.2	15.5	38.8	180+	
diethylamine	7.8	8.6	17.5	58.7	75.8	180+

The water-soluble monoethanolamine, triethanolamine and diethylamine salts of sulfobenzoic acid prepared as described above were tested for detergency by the method described by Jay C. Harris and Earl L. Brown in the Journal of the American Oil Chemists' Society, vol. 27, p. 135–143 (1950). By the application of this method it is possible to evaluate the detergency of a product in terms of any standard detergent. For convenience I have compared the detergency of the present and related products with the detergency of Gardinol WA, which is a commercial detergent produced by sulfating the alcohols derived by hydrogenation of coconut oil fatty acids. The following results were obtained for tests conducted in water of 50 p. p. m. hardness:

Compound Tested	Percent Detergency
monoethanolamine	121
triethanolamine	107
diethylamine	113

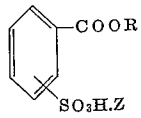
Evaluation of the foaming properties of the monoethanolamine and triethanolamine salts by the Ross Miles Lathering Test (a proposed method of the American Society for Testing Materials) gave the following values in water hardness of 50 p. p. m.:

Lather heights, cm.

Salt of Tridecyl Sulfobenzoate Tested	At Once	After 5 Minutes
monoethanolamine	15.0	14.7
triethanolamine	14.7	14.5

What I claim is:

1. Surface-active agents having the formula



in which R is a primary alkyl radical having an average of 13 carbon atoms derived from an alcohol formed by the carbonylation and hydrogenation of an olefin selected from the class consisting of triisobutylene and tetrapropylene and mixtures of the same and in which Z is selected from the class consisting of mono-, di- and trialkylamines in which the alkyl radicals contains from 1 to 12 carbon atoms, mono-, di- and trialkanolamines in which the alkanol radical has from 1 to 4 carbon atoms and alkylenepolyamines and polyalkylenepolyamines having from 2 to 4 amino groups in the molecule and from 2 to 3 carbon atoms in the alkylene chain, and morpholine.

2. The monoethanolamine salt of the tridecyl ester of sulfobenzoic acid.

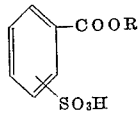
3. The triethanolamine salt of the tridecyl ester of sulfobenzoic acid.

4. The n-butylamine salt of the tridecyl ester of sulfobenzoic acid.

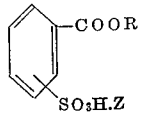
5. The 2-ethylhexylamine salt of the tridecyl ester of sulfobenzoic acid.

6. The diethylenetriamine salt of the tridecyl ester of sulfobenzoic acid.

7. The process of preparing surface-active agents which comprises contacting a sulfobenzoate having the formula



wherein R is a primary alkyl radical having an average of 13 carbon atoms derived from an alcohol formed by the carbonylation and hydrogenation of an olefin selected from the class consisting of triisobutylene, tetrapropylene, and mixtures thereof with an amine compound selected from the class consisting of mono-, di- and trialkylamines in which the alkyl radical contains from 1 to 12 carbon atoms, mono-, di- and trialkanolamines in which the alkanol radical has from 1 to 4 carbon atoms and alkylenepolyamines and polyalkylenepolyamines having from 2 to 4 amino groups in the molecule and from 2 to 3 carbon atoms in the alkylene chain and morpholine, and recovering from the resulting reaction product salts having the formula



in which R is a primary alkyl radical having an average of 13 carbon atoms derived from an alcohol formed by the carbonylation and hydrogenation of an olefin selected from the class consisting of triisobutylene, tetrapropylene and mixtures thereof and Z is selected from the class consisting of mono-, di- and trialkylamines in which the alkyl radical contains from 1 to 12 carbon atoms, mono-, di- and trialkanolamines in which the alkanol radical has from 1 to 4 carbon atoms and alkylenepolyamines and polyalkylenepolyamines having from 2 to 4 amino

groups in the molecule and from 2 to 3 carbon atoms in the alkylene chain, and morpholine.

8. The process which comprises contacting tridecyl sulfobenzoate with monoethanolamine and recovering the monoethanolamine salt of tridecyl sulfobenzoate from the resulting reaction product.

9. The process which comprises contacting tridecyl sulfobenzoate with triethanolamine and recovering the triethanolamine salt of tridecyl sulfobenzoate from the resulting reaction product.

10. The process which comprises contacting tridecyl sulfobenzoate with n-butylamine and recovering the n-butylamine salt of tridecyl sulfobenzoate from the resulting reaction product.

11. The process which comprises contacting tridecyl sulfobenzoate with 2-ethylhexylamine and recovering the 2-ethylhexylamine salt of tridecyl sulfobenzoate from the resulting reaction product.

12. The process which comprises contacting tridecyl sulfobenzoate with diethylenetriamine and recovering the diethylenetriamine salt of tridecyl sulfobenzoate from the resulting reaction product.

No references cited.