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3,412,155

FATTY BRANCHED AMINE DIOXIDES

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No Drawing. Continuation-in-part of application Ser. No. 402,921, Oct. 9, 1964. This application Sept. 15, 1965, Ser. No. 487,606

7 Claims. (Cl. 260—583)

ABSTRACT OF THE DISCLOSURE

Fatty branched amine dioxides made by oxidizing the fatty diamine precursor with a strong oxidizing agent such as H_2O_2 . These are useful as detergents.

This application is a continuation-in-part application of Ser. No. 402,921 filed Oct. 9, 1964, now abandoned.

This invention relates to novel derivatives of fatty acid and more particularly to a novel class of fatty amine dioxides which are particularly useful as detergents for dishwashing.

Roe and Swern of the U.S. Department of Agriculture, in the Journal of the American Oil Chemists Society periodical, volume 37, pages 661 to 668 (1960) report that unsaturated fatty acids may be carboxylated with carbon monoxide. The monoxide adds to the double bond to create a long chain fatty acid with two or more reactive groups therein. With two or more reactive groups, the fatty compound not only has good hydrophobic characteristics, but also has high potency as a detergent with the proper reactive groups in place of the carboxy group. Unfortunately, no further work was apparently done by Roe and Swern or others along this line.

Accordingly, an object of this invention is to provide novel difunctional fatty compounds which are especially useful as detergents.

Another object is to provide a novel class of fatty amine dioxides.

Still another object is to provide novel methods for the manufacture of such fatty amine dioxides.

A further object is to provide a novel class of compounds which are good detergents especially in dishwashing machines.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the several steps and the relation of one or more of such steps with respect to each of the others, and the compound possessing the features, properties, and the relation of elements, which are exemplified in the following detailed disclosure, and the scope of the invention will be indicated in the claims.

It has now been found that a novel class of fatty amine dioxides may be prepared by oxidizing the fatty amine precursor with a strong oxidizing agent. For example, by reacting N,N,N',N' -tetramethylaminomethylstearylamine with hydrogen peroxide, N,N,N',N' -tetramethylaminomethylstearylamine dioxide is obtained. Or, by reacting ethoxylated aminomethylstearylamine with hydrogen peroxide, its dioxide will be obtained.

Reference is now made to the following examples which illustrate the synthesis of a particular fatty amine dioxide:

EXAMPLE I

N,N,N',N' -tetramethylaminomethylstearylamine dioxide

A 250 ml., three neck flask, equipped with a magnetic stirrer, thermometer, addition funnel and reflux condenser is charged with N,N,N',N' -tetramethylaminometh-

2

ylstearylamine (17.2 gms., 0.0485 mole) and 15 gms. 50% aqueous isopropanol. Hydrogen peroxide (8.2 gms., 45.5% aqueous, 0.11 mole) is added dropwise at 30–45° C. over a period of thirty-five minutes. The reaction mixture is stirred overnight at about 30° C. resulting in a colorless solution.

Analysis.—Amine oxide, 43.6%; free amine, 2%; free peroxide, 3.26%.

EXAMPLE II

Ethoxylated aminomethylstearylamine dioxide

A 250 ml., three neck flask, equipped with a magnetic stirrer, thermometer, addition funnel and condenser is charged with 57.4 gms. (0.102 mole, 0.204 equivalent) of ethoxylated aminomethylstearylamine (Neutralization Equivalent 282), 17.6 gms. of water and 30.5 gms. of isopropanol. Hydrogen peroxide (16.9 gms., 0.244 mole, 49% active) is then added dropwise over a period of four hours at 30–45° C. The reaction mixture is then allowed to stir at room temperature for an additional 67 hours. An amber solution will be obtained having the following analysis:

Analysis.—Amine oxide, 48.5%; free amine, nil; free peroxide, 0.36%.

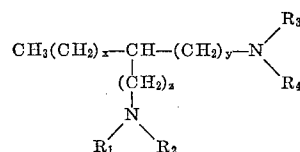
EXAMPLE III

Ethoxylated aminostearylamine dioxide

A 250 ml., three neck flask, equipped with a magnetic stirrer, thermometer, addition funnel and condenser is charged with 92.1 gms. (0.181 mole, 0.362 equivalent) of ethoxylated aminostearylamine (Neutralization Equivalent 254), 26.5 gms. of water and 49.1 gms. of isopropanol. Hydrogen peroxide (30.2 gms., 0.435 mole, 49% active) is then added dropwise over a period of four hours at 30–45° C. The reaction mixture is then stirred at room temperature for an additional 65–70 hours. An amber solution will be obtained having the following analysis:

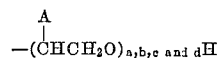
Analysis.—Amine oxide, 50.7%; free amine, nil; free peroxide, 0.11%.

Among the aminofatty amines that may be treated in accordance with the method of this invention, those falling within the following formula are operable:

Formula I

where

$R_1, R_2, R_3, R_4 = \text{CH}_3-$, CH_3CH_2- ,



and mixtures thereof

$\text{A} = \text{H}-$, CH_3-

a, b, c , and $d = 1$ to 50

$x+y = 6$ to 22

$z = 0$ to 2

Among specific compounds falling within the above formula are the N,N,N',N' -tetramethyl derivatives of aminocaprylamine, aminocaprylamine, aminolaurylamine, aminomyristylamine, aminopalmitylamine, aminostearylamine, aminoarachidylamine, aminobehenylamine, aminolignocerylamine, aminomethylcaprylamine, aminomethylcaprylamine, aminomethylaurylamine, aminomethylmyristylamine, aminomethylpalmitylamine, aminomethylstearylamine, aminomethylarachidylamine, aminomethylbehenylamine, aminomethylignocerylamine,

3

aminoethylcaprylylamine, aminoethylcaprylamine, aminoethylaurylamine, aminoethylmyristylamine, aminoethylpalmitylamine, aminoethylstearylamine, aminoethylarachidylamine, aminoethylbehenylamine, aminoethyl-lignocerylamine or mixtures thereof such as derivable from natural fats and oils, such as tallow, soybean oil and coconut oil; the alkoxylated (ethoxylated and propoxylated) derivatives of aminocaprylylamine, aminocaprylamine, aminolaurylamine, aminomyristylamine, aminopalmitylamine, aminostearylamine, aminoarachidylamine, aminobehenylamine, aminolignocerylamine, aminomethylcaprylylamine, aminomethylcaprylamine, aminomethylaurylamine, aminomethylmyristylamine, aminomethylpalmitylamine, aminomethylstearylamine, aminomethylarachidylamine, aminomethylbehenylamine, aminomethyl-lignocerylamine, aminoethylcaprylylamine, aminoethylcaprylamine, aminoethylaurylamine, aminoethylmyristylamine, aminoethylpalmitylamine, aminoethylstearylamine, aminoethylarachidylamine, aminoethylbehenylamine, aminoethyl-lignocerylamine or mixtures thereof such as derivable from natural fats and oils, such as tallow, soybean oil and coconut oil.

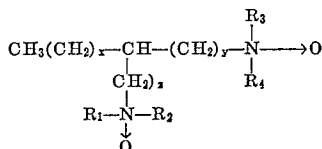
The strong oxidizing agent that is useful, falls within a relatively wide scope of compounds. Among such are hydrogen peroxide, perbenzoic acid and Caro's acid.

Usually between 2.0 to 20.0 molar equivalent percent in excess of the fatty amine utilized is used, and the preferred excess amount is 5.0 molar equivalent percent.

When an alkoxylated amine is utilized, it may have been alkoxylated with from 4 to 200 moles of an alkylene oxide, such as ethylene oxide, or propylene oxide.

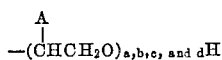
The fatty amine dioxides that may be formed by the method of this invention are those which fall within the following formula:

Formula II



where

$\text{R}_1, \text{R}_2, \text{R}_3, \text{R}_4 = \text{CH}_3-, \text{CH}_3\text{CH}_2-,$



and mixtures thereof

$\text{A} = \text{H}-, \text{CH}_3-$

$a, b, c, \text{ and } d = 1 \text{ to } 50$

$x+y = 6 \text{ to } 22$

$z = 0 \text{ to } 2$

Among compounds which fall within the above formula are the N,N,N',N'-tetramethyl derivatives of

aminocaprylylamine dioxide,
aminocaprylamine dioxide,
aminolaurylamine dioxide,
aminomyristylamine dioxide,
aminopalmitylamine dioxide,
aminostearylamine dioxide,
aminoarachidylamine dioxide,
aminobehenylamine dioxide,
aminolignocerylamine dioxide,
aminomethylcaprylylamine dioxide,
aminomethylcaprylamine dioxide,
aminomethylaurylamine dioxide,
aminomethylmyristylamine dioxide,
aminomethylpalmitylamine dioxide,
aminomethylstearylamine dioxide,
aminomethylarachidylamine dioxide,
aminomethylbehenylamine dioxide,
aminomethyl-lignocerylamine dioxide,
aminoethylcaprylylamine dioxide,

4

aminoethylcaprylamine dioxide,
aminoethylaurylamine dioxide,
aminoethylmyristylamine dioxide,
aminoethylpalmitylamine dioxide,
aminoethylstearylamine dioxide,
aminoethylarachidylamine dioxide,
aminoethylbehenylamine dioxide,
aminoethyl-lignocerylamine dioxide

or mixtures thereof such as derivable from natural fats and oils, such as tallow, soybean oil and coconut oil; and ethoxylated or propoxylated (4 to 200 moles) of

aminocaprylylamine dioxide,
aminocaprylamine dioxide,
aminolaurylamine dioxide,
aminomyristylamine dioxide,
aminopalmitylamine dioxide,
aminostearylamine dioxide,
aminoarachidylamine dioxide,
aminobehenylamine dioxide,
aminolignocerylamine dioxide,
aminomethylcaprylylamine dioxide,
aminomethylcaprylamine dioxide,
aminomethylaurylamine dioxide,
aminomethylmyristylamine dioxide,
aminomethylpalmitylamine dioxide,
aminomethylstearylamine dioxide,
aminomethylarachidylamine dioxide,
aminomethylbehenylamine dioxide,
aminomethyl-lignocerylamine dioxide,
aminoethylcaprylylamine dioxide,
aminoethylcaprylamine dioxide,
aminoethylaurylamine dioxide,
aminoethylmyristylamine dioxide,
aminoethylpalmitylamine dioxide,
aminoethylstearylamine dioxide,
aminoethylarachidylamine dioxide,
aminoethylbehenylamine dioxide,
aminoethyl-lignocerylamine dioxide

or mixtures thereof such as derivable from natural fats and oils, such as tallow, soybean oil and coconut oil.

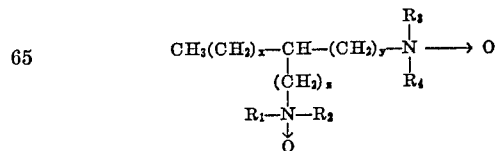
The above compounds are useful as surfactants and especially as dishwashing detergents.

It will thus be seen that the objects set forth above, among those made apparent from the preceding description, are efficiently attained and, since certain changes may be made in carrying out the above method and in the compounds set forth without departing from the scope of the invention, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

It is also to be understood that the following claims are intended to cover all of the generic and specific features of the invention herein described, and all statements of the scope of the invention, which, as a matter of language, might be said to fall therebetween.

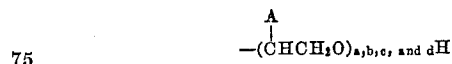
Now that the invention has been described, what is claimed as new and desired to be secured by Letters Patent is:

1. The fatty amine dioxide falling within the following formula:



where

$\text{R}_1, \text{R}_2, \text{R}_3, \text{R}_4 = \text{CH}_3-, \text{CH}_3\text{CH}_2-,$



5

and mixtures thereof

A=H—, CH₃—

a, b, c, and d=1 to 50

x+y=6 to 22

z=0 to 2

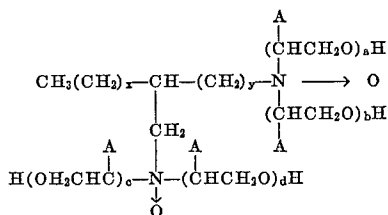
2. N,N,N',N'-tetramethylaminomethylstearylamine dioxide.

3. N,N,N',N'-tetramethylaminoundecylamine dioxide.

4. A mixture of N,N,N',N'-tetramethylaminomethylfattyamine dioxides the fatty constituent being derived from the natural fats and oils.

5. A mixture of N,N,N',N'-tetramethylaminomethylfattyamine dioxides the fatty constituent being derived from tallow.

6. The alkoxyated aminomethylstearylamine dioxides falling within the following formula:



where

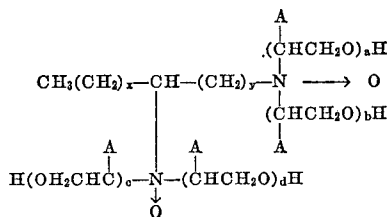
A=H—, CH₃—

a, b, c, d=1 to 50

x+y=6 to 22

6

7. The alkoxyated aminostearylamine dioxides falling within the following formula:



where

A=H—, CH₃—

a, b, c, d=1 to 50

x+y=6 to 22

References Cited

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CHARLES B. PARKER, *Primary Examiner*.R. L. RAYMOND, *Assistant Examiner*.