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IODINE DETERGENT COMPOSITIONS

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7 Claims. (Cl. 252—106)

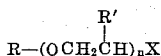
As is well known in the art, liquid nonionic polyglycol ether type surface active agents obtained by condensing alkaline oxides with water insoluble organic compounds containing at least 6 carbon atoms and having an active hydrogen are capable of readily dissolving iodine to yield germicidal-detergent compositions. After appropriate compounding with water and a sufficient amount of acid to obtain a pH of at least about 5 or lower, these compositions have been shown to be particularly effective for use in various apparatus for washing milk bottles, beer bottles and like containers for eatable products and for cleaning apparatus and equipment concerned in the manufacture and transport of such products. However, although these compositions possess excellent detergent and germicidal characteristics, they suffer from the disadvantage of producing a relatively high level of foam which detracts from their overall efficiency and economy in commercial cleaning and sanitizing operations. It should be immediately apparent that excessive foaming in a cleaning compound affects its ability to adequately clean the container being so treated and also poses a potential danger to the various mechanical equipment employed in commercial cleaning and sanitizing operations.

Accordingly, it is the primary object of this invention to provide a novel iodine detergent composition possessing the desirable germicidal properties and detergent characteristics of the prior art compositions formulated from liquid nonionic polyglycol ether type surfactants but without the disadvantage of having excessive foaming characteristics.

It is still another object of this invention to provide a novel composition of matter comprising iodine and a chlorine containing nonionic surface active composition having excellent germicidal-detergent characteristics as well as low foaming characteristics.

It has now been found that the above objectives can be obtained by formulating a germicide-detergent composition from iodine and various halide containing non-ionic surface active compositions. Quite unexpectedly, it has been found that if certain halide containing nonionic surface active agents are employed for formulating the novel compositions of this invention a composition results which has extremely low foaming properties along with the expected germicide and detergent properties.

The nonionic surface active agents which are employed to prepare the novel compositions of this invention are halides of polyethoxylated phenols and polyethoxylated aliphatic alcohols characterized by the following general formula:



wherein R represents either an alkyl group containing 8 to 22 carbon atoms or a phenyl or naphthyl substituted in the ring with 1, 2 or 3 alkyl groups having a total of from 6 to 36 carbon atoms; R' represents hydrogen, methyl, or ethyl; *n* represents a positive integer of from

2 to 12 inclusive and X represents chlorine, bromine or iodine. The above compositions are known in the art and are generally prepared by converting any polyethoxylated phenol or polyethoxylated aliphatic alcohol to the corresponding halide by conventional means. The preparation of polyethoxylated phenols and polyethoxylated aliphatic alcohols upon which the above halides are based are described in great detail in U.S. Patents 1,970,578, 2,213,477, 2,575,832, 2,593,112, and 2,676,975. The corresponding halides and methods for their preparation are disclosed in U.S. Patents 2,249,111, 2,098,203, 2,097,441, 2,097,411, 2,209,911 and 3,061,552, the complete disclosures and teachings of which are incorporated herein by reference thereto.

15 The novel compositions of this invention are prepared simply by adding elemental iodine to the halogen containing surfactants above described at a temperature ranging from about 20° C. to 100° C. and agitating for periods of times ranging from 10 to 20 minutes. However, particularly effective results have been obtained when the
20 elemental iodine was added at temperatures ranging from 40° C. to 80° C. and agitation was carried out for periods of time ranging from 2 to 6 hours.

25 The amount of elemental iodine which is added to the halides of polyethoxylated phenols and polyethoxylated aliphatic alcohols is not narrowly critical but it is preferred to add sufficient iodine so as to provide from 1 to 30% by weight of available iodine with from 10 to 22% by weight available iodine being particularly preferred.

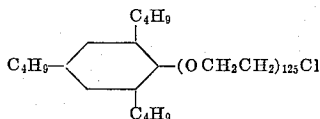
30 Germicidal cleansing solutions may be formulated from the iodine containing surface active agents in a conventional manner, most frequently by the addition of an acid such as phosphoric acid or hydrochloric acid and water.

As has heretofore been pointed out, the novel compositions of this invention are prepared by adding elemental iodine to a specific class of halides of polyethoxylated phenols and polyethoxylated aliphatic alcohols. It should be immediately apparent that the surface active agents employed to formulate the novel compositions of the instant invention differ from the polyethoxylated phenols and polyethoxylated aliphatic alcohols heretofore proposed not only by the fact that they have a terminal halogen atom in place of the terminal hydroxyl group of the prior art compounds, but also due to the fact that the compositions have only from 2 to 12 recurring oxyalkylene units. It should be pointed out that if halogen-containing nonionic surface active agents are employed having more than 12 recurring oxyalkylene units, compositions will be obtained which have effective detergent and germicidal properties as would be expected but that these compositions will have no better foaming properties than similar compositions prepared from nonionic surface active agents containing terminal hydroxyl groups. Therefore, the heart of the instant invention involves the use of nonionic surface active agents having terminal halide atoms and from 2 to 12 recurring oxyalkylene units.

60 The examples will now illustrate the best mode contemplated for carrying out the invention but it is to be understood that it is not intended to be limited thereto.

Example 1

One hundred forty-four parts by weight of a halogen-
65 ated surface active agent of the following formula:



and 56 parts by weight of iodine were mixed together at

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100° C. and agitated for two hours to yield 200 parts by weight of a composition having 21.9 weight percent of available iodine.

0.10% by weight of the resulting composition was then dissolved in water and placed in a graduated cylinder and agitated in a uniform manner. The foam height was then measured and a comparison was made with the foam height produced by the iodine derivative of the corresponding non-halogenated surfactant with the following results:

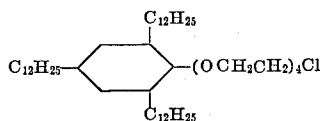
Foam height of halogenated base, millimeters ----- 20
Foam height of non-halogenated base, millimeters -- 22

From the above it can be seen that there is no substantial difference in foam height when using a halogenated as opposed to a non-halogenated surface active agent. It can be seen that in this instance the number of recurring oxyalkylene units was 125.

Example 2

One hundred forty-four parts by weight of a halogenated surface active agent. It can be seen that in this instance the number of recurring oxyalkylene units was 125.

One hundred forty-four parts by weight of a halogenated surface active agent of the following formula:



and 56 parts by weight of iodine were mixed together at 90° C. for 0.5 hour to yield 200 parts by weight of the product having 22.3 weight percent of available iodine.

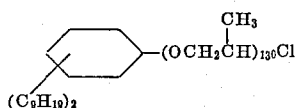
A foam comparison between the product obtained according to the above procedure and the corresponding non-halogenated base was carried out in the exact manner as set forth in Example 1 with the following results:

Foam height of halogenated base, millimeters ----- 8
Foam height of non-halogenated base, millimeters -- 26

From the above example it can be seen that more than a 300% reduction in foam occurred by using the corresponding halide derivative. It is also pointed out that in this example the composition employed had 4 recurring oxyethylene units.

Example 3

One hundred seventy-four parts by weight of a halogenated nonionic surface active agent of the formula:



and 26 parts by weight of iodine were mixed together at 80° C. and agitated for four hours to yield 200 parts by weight of a composition having 10.1% available iodine.

Comparison of the above composition with the corresponding non-halogenated derived material in accordance with the procedure of Example 1 yielded the following results:

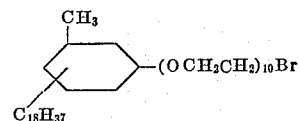
Foam height of halogenated base, millimeters ----- 24
Foam height of non-halogenated base, millimeters -- 22

From the above results it can be seen that in this instance the foam produced was even greater than the foam resulting from the corresponding non-halogenated base. It can be seen that the composition employed in this example had 130 recurring oxyalkylene units.

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Example 4

Two hundred parts by weight of a halogenated nonionic surface active agent of the following formula:



and 85 parts by weight of iodine were mixed together at 70° C. and agitated for 12 hours to yield 285 parts by weight of a composition having 23.8% of available iodine.

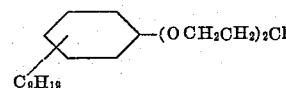
Comparison with the composition made from the corresponding non-halogenated surface active agent in accordance with the procedure of Example 1 had the following results:

Foam height of halogenated base, millimeters ----- 7
Foam height of non-halogenated base, millimeters --- 28

From the above it can be seen that almost a 400% reduction in foam occurred when using the halogenated nonionic surface active agent. It is also pointed out that in this instance there were present 10 recurring oxyethylene units in the halogenated base.

Example 5

One hundred fifty-two parts by weight of a halogenated nonionic surface active agent of the following formula:



and 48 parts by weight of iodine were mixed together at 60° C. and agitated for 3 hours to yield 200 parts by weight of a composition having 16.2% of available iodine.

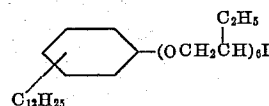
Comparison of foam with an iodine composition prepared from the corresponding non-halogenated base yielded the following results:

Foam height of halogenated base, millimeters ----- 10
Foam height of non-halogenated base, millimeters --- 30

From the above results it can be seen that the 200% reduction in foam occurred in this instance.

Example 6

One hundred sixty-five parts by weight of a halogenated nonionic surface active agent of the following formula:



and 58 parts by weight of iodine were mixed at 50° C. and agitated for 4 hours to yield 223 parts by weight of a composition having 20.0% of available iodine.

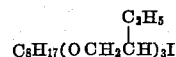
Comparison of the foam produced by this composition with one prepared from the corresponding non-halogenated base yielded the following results:

Foam height of halogenated base, millimeters ----- 8
Foam height of non-halogenated base, millimeters --- 25

From the above it can be seen that again more than a 300% reduction in foam occurred when using the halogenated base.

Example 7

Two hundred parts by weight of a halogenated nonionic surface active agent of the formula:



and 80 parts by weight of iodine were mixed together at 40° C. and stirred for 10 hours to yield 280 parts by weight of a composition having 24.8% of available iodine.

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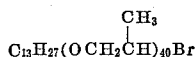
Comparison of the foaming characteristics of the above product with one prepared from the corresponding non-halogenated base yielded the following results:

Foam height of halogenated base, millimeters ----- 10
Foam height of non-halogenated base, millimeters --- 20

From the above example it can be seen that a 200% reduction in foam occurred.

Example 8

Two hundred parts by weight of a halogenated nonionic surface active agent of the following formula:



and 30 parts by weight of iodine were mixed together at 30° C. and stirred for 6 hours to yield 230 parts by weight of a composition having 11.4% of available iodine.

Comparison of the foam characteristics of the above product with the one prepared from the corresponding non-halogenated base had the following results:

Foam height of halogenated base, millimeters ----- 28
Foam height of non-halogenated base, millimeters --- 26

From the above results it can be seen that in this instance the iodine composition prepared from the halogenated nonionic surface active agent produced more foam than the one prepared from the resulting non-halogenated base. It should be noted that in this instance the composition contained 40 recurring oxyalkylene units.

Example 9

Two hundred parts by weight of a halogenated nonionic surface active agent of the following formula:



and 56 parts by weight of iodine were mixed together at 20° C. and stirred for 5 hours to yield 256 parts by weight of a composition having 16.4% available iodine.

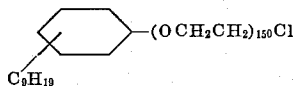
Comparison of the foaming characteristics of the above product with one prepared from the corresponding non-halogenated base yielded the following:

Foam height of halogenated base, millimeters ----- 30
Foam height of non-halogenated base, millimeters --- 32

From the above data it can be seen that the compound prepared from the halogenated nonionic agent had substantially the same foaming characteristics as one prepared from the non-halogenated surface active agent. It should be noted that in this instance the nonionic surface active agent employed had 120 recurring oxyalkylene units.

Example 10

One hundred forty-four parts by weight of a nonionic surface active agent of the following formula:



and 56 parts by weight of iodine were mixed at 80° C. and stirred for 6 hours to yield 200 parts by weight of a composition having 24.6% available iodine.

Comparison of foaming characteristics of the above composition with one prepared from the corresponding non-halogenated base yielded the following results:

Foam height of halogenated base, millimeters ----- 32
Foam height of non-halogenated base, millimeters --- 36

From the above example, it can again be seen that in this instance the results obtained from the halogenated derivative were substantially the same as the non-halogenated one. It is again noted that the halogenated

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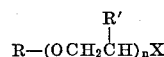
nonionic surface active agent employed in this example had 150 recurring oxyalkylene units.

Many modifications and variations of the above examples will occur to those skilled in the art such as employing compounds mentioned in the specification for those mentioned in the specific examples. Therefore, it is not intended that this invention be limited except as necessitated by the appended claims.

What is claimed is:

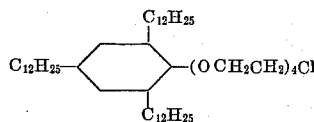
1. A water-soluble, low-foaming germicidal-detergent composition consisting essentially of (A) the halide of a nonionic surface active agent selected from the class consisting of polyethoxylated phenols and polyethoxylated aliphatic alcohols having from 2 to 12 recurring oxyalkylene units and, (B) elemental iodine in an amount sufficient to provide from about 1 to 30% by weight available iodine in said composition.

2. The composition as defined in claim 1 wherein the halide of the nonionic surface active agent has the formula:

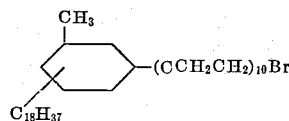


wherein R represents a radical selected from the group consisting of alkyl radicals of from 8 to 22 carbon atoms, phenyl and naphthyl radicals substituted with from 1 to 3 alkyl groups having a total of from 6 to 36 carbon atoms, R' is selected from the group consisting of hydrogen, methyl and ethyl, n represents a positive integer of from 2 to 12 and X represents a halogen selected from the group consisting of chlorine, bromine and iodine.

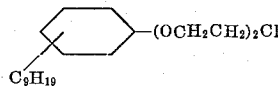
3. The composition as defined in claim 1 wherein the halide of the nonionic surface active agent has the formula:



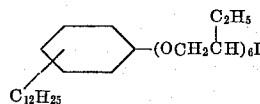
4. The composition as defined in claim 1 wherein the halide of the nonionic surface active agent has the formula:



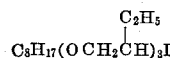
5. The composition as defined in claim 1 wherein the halide of the nonionic surface active agent has the formula:



6. The composition as defined in claim 1 wherein the halide of the nonionic surface active agent has the formula:



7. The composition as defined in claim 1 wherein the halide of the nonionic surface active agent has the formula:



References Cited by the Examiner

UNITED STATES PATENTS

2,209,911	7/1940	Bruson et al. -----	260—613
2,212,536	8/1940	Britton et al. -----	260—613
2,997,421	8/1961	Hosmer et al. ----	252—106 XR
3,029,183	4/1962	Winicov et al. -----	260—613
3,061,552	10/1962	Schenck et al. ----	252—156 XR

FOREIGN PATENTS

703,091 1/1954 Great Britain.

LEON D. ROSDOL, *Primary Examiner.*JULIUS GREENWALD, *Examiner.*A. T. MEYERS, *Assistant Examiner.*

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

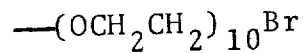
Patent No. 3,277,010

October 4, 1966

Leslie M. Schenck et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, lines 22 to 24, strike out "One hundred forty-four parts by weight of a halogenated surface active agent. It can be seen that in this instance the number of recurring oxyalkylene units was 125."; column 6, line 48, the right-hand portion of the formula should read as shown below instead of as in the patent:



Signed and sealed this 5th day of September 1967.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents