

1

2,809,937

GERMICIDAL DETERGENT COMPOSITION

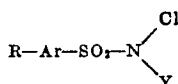
Frederick William Gray, Belleville, N. J., assignor to Colgate-Palmolive Company, Jersey City, N. J., a corporation of Delaware

No Drawing. Application February 26, 1954,
Serial No. 412,937

2 Claims. (Cl. 252—106)

The present invention relates to germicidal detergent compositions containing N-chlorinated higher alkyl aryl sulfonamide compounds.

In accordance with the present invention, a germicidal detergent composition consists essentially of from about 5 to about 95% of a water-soluble organic anionic detergent and from about 0.01 to 50% of a sulfonamide having the formula:



wherein R is an alkyl group of about 10 to 15 carbon atoms, Ar is a benzene nucleus, and Y is selected from the group consisting of chlorine, hydrogen, alkali metal, and alkaline earth metal.

The alkyl radical on the aromatic nucleus of the sulfonamide compounds of the present composition is straight or branched chain and contains about 10 to 22 carbon atoms. Suitable examples of such radicals are decyl, dodecyl, keryl, pentadecyl, hexadecyl, mixed long chain alkyls derived from long chain fatty materials, cracked paraffin wax olefins, polymers of lower mono-olefins, etc. It is preferred that the alkyl substituent average about 12 to about 18 carbon atoms. For example, propylene may be polymerized to the tetramer and condensed with benzene in the presence of an alkylation catalyst, e. g. a Friedel-Crafts catalyst, to yield a mixture comprising essentially the dodecyl benzene derivative which is suitable for preparation of the desired sulfonamide type compounds.

The aromatic nucleus of the sulfonamide compounds of the present composition may be mono or polynuclear and may contain other substituents which do not interfere with the character of the chlorinated product. Thus a variety of suitable aromatic nuclei may be derived from benzene, naphthalene, anthracene, toluene, xylene, phenol, cresols, etc., benzene being a preferred embodiment of the invention as exemplified by dodecyl benzene sulfonchloramide, keryl benzene sulfonchloramide and decyl benzene sulfonchloramide. Other suitable materials are keryl toluene sulfonchloramide, dodecyl naphthalene sulfonchloramide, decyl anthracene sulfonchloramide, and pentadecyl ethylbenzene sulfonchloramide.

The alkylated aromatic nucleus preferably carries one N-chlorinated sulfonamide substituent, such as, for example, dodecyl benzene monosulfonchloramide, keryl toluene monosulfonchloramide, and pentadecyl naphthalene monosulfonchloramide. However, it is also within the scope of the invention to have poly substitution, such as alkyl aryl di- and trisulfonchloramides, suitable examples thereof being dodecyl naphthalene disulfonchloramide and keryl naphthalene trisulfonchloramide. In particular, the higher alkyl benzene monosulfonmonochloramides are

2

preferred, examples thereof comprising decylbenzene monosulfonmonochloramide, keryl benzene monosulfonmonochloramide, dodecylbenzene monosulfonmonochloramide, tetrapropylene benzene monosulfonmonochloramide, and the alkali metal salts thereof such as the sodium, potassium and lithium salts and alkaline earth metal salts such as the calcium and magnesium salts. Other suitable sulfonamide compounds are: dodecylbenzene monosulfondichloramide, keryl naphthalene disulfonmonochloramide, pentadecyl toluene monosulfonmonochloramide and the above-indicated salts.

The instant sulfonamide compounds may be prepared in any convenient manner, starting with the appropriate long-chain alkyl aryl hydrocarbon or its sulfonic acid derivative. Using the alkyl aryl hydrocarbon as an initial reactant, the suitable sulfonic acid derivative may be formed by any convenient sulfonation procedure, such as reaction with oleum or chlorosulfonic acid. These alkyl aryl sulfonic acid compounds are well known in the art.

The appropriate aforesaid long chain sulfonic acid may readily be converted to an amide by treatment with ammonia. The sulfonchloramide derivative may then be produced by suspending or dissolving the said sulfonamide in an aqueous alkaline medium and then passing chlorine gas in excess of the stoichiometrically required amount through the mixture while subjecting the mixture to agitation and maintaining it at a slightly elevated temperature of about 40° C. to 50° C. The chlorine reacts rapidly with the amide group and replaces at least one of the replaceable hydrogen atoms attached thereto, the product being a solid precipitate which may be recovered by any means suitable for solid-liquid phase separation, including gravity settling, decantation, filtration, centrifuging, etc. The product is recovered in the form of its salt depending upon the base used in the alkaline medium. Any suitable alkali metal (e. g. sodium and potassium hydroxide) or alkaline earth metal base (e. g. calcium and magnesium hydroxide) may be used to form the corresponding salt. The free N-chlorinated sulfonamide may be prepared from the corresponding salt in any suitable manner, such as reacting the suitable salt with mineral acid and separating the inorganic salt formed as a by-product from the formed N-chlorosulfonamide compound. The dichlorinated derivative may be prepared by further chlorination under similar conditions.

The purified sulfonchloramide compounds and their salts may be dried, washed, recrystallized or subjected to other conventional means of purification suitable to such materials.

More specifically, suitable N-chlorinated higher alkyl aryl sulfonamides may be prepared as follows.

About 736 parts by weight of 96.5% sulfuric acid are placed in a vessel equipped with an agitator and 435 parts of alkyl benzene (prepared by alkylating benzene with a propylene tetramer) are added. The vessel is cooled so as to maintain the reactants at 20° C. to 30° C. On complete addition of the hydrocarbon and while maintaining the temperature at about 20° C., 1062 parts of chlorsulfonic acid are slowly added over a period of about 2 hours. The reaction takes place with a strong evolution of hydrogen chloride. The contents of the vessel are agitated throughout the entire run and for two hours after all the chlorsulfonic acid has been added, after which the mixture is allowed to settle for several hours. The upper layer contains the alkylbenzene sulfonyl chloride in approximately 90% yield, and is removed for further processing.

About 290 parts of the crude alkylbenzene sulfonyl

3

chloride are introduced dropwise into 450 parts of 28% ammonium hydroxide while the reaction mixture is thoroughly agitated and the temperature maintained at 25° C. to 30° C. The sulfonamide separates as a whitish yellow oil. On ether extraction, the sulfonamide is obtained as a pale yellow viscous oil. The yield is about 85% of the theoretical yield based on the alkyl benzene employed.

About 150 parts of the alkylbenzene sulfonamide previously prepared are mixed in a vessel with 1000 parts of 10% sodium hydroxide. While the liquids are agitated, about 45 parts of chlorine gas is bubbled into the mixture continuously over a period of 1½ hours. The reactants are maintained at 40° C. to 50° C. A voluminous white precipitate is formed which is allowed to settle, the supernatant liquid is decanted, and the precipitate is slurried in cold water and filtered. The product is recrystallized from aqueous ethanol to yield a dry, stable, white material which foams readily and has a characteristic hypochlorite odor in an aqueous medium. The yield of the product, which is the monosodium salt of the monosulfonmonochloramide of the alkyl benzene employed is about 80% of the theoretical yield. The product analyzes to an active or available chlorine content of 8.4%, a total chlorine content of 9.29%. A sodium salt of a monosulfonmonochloramide of an alkyl benzene derived from a propylene tetramer and having a calculated molecular weight of 240 has a theoretical calculated chlorine content of 9.4%.

The previously described process is repeated using potassium hydroxide in the place of sodium hydroxide in the chlorination step. The product is a potassium salt of the monosulfonmonochloramide of the alkyl benzene starting material.

The previously described process is repeated using calcium hydroxide in the place of sodium hydroxide in the chlorination step. The product is a calcium salt of the monosulfonmonochloramide of the alkyl benzene starting material.

The instant sulfanamide compounds have particular utility in the form of detergent compositions exhibiting superior germicidal bleaching and deterative power. It has been found that they are compatible with suitable detergents resulting in a product having excellent foaming and bleaching action in a variety of detergent applications. These chlorinated sulfonamides may be admixed with detergents selected from the group consisting of water-soluble organic anionic and non-ionic detergents in any suitable proportion. Among the suitable anionic detergents are the water-soluble higher fatty acid soaps (e. g. sodium laurate, potassium stearate, sodium salts of higher fatty acids derived from coconut oil, palm oil and tallow), and the sulfated and sulfonated-organic detergents. Examples of the latter are water-soluble salts of higher alkyl aryl sulfonate (e. g. sodium dodecylbenzenesulfonate, sodium kerylbenzenesulfonate), higher alkyl sulfate detergent (e. g. sodium lauryl sulfate, potassium cetyl sulfate), sulfuric acid esters of polyhydric alcohols incompletely esterified with higher fatty acids (e. g. sodium coconut monoglyceride monosulfate), sulfated higher fatty acid amides (e. g. sodium lauric monoethanolamide sulfate) etc. Among the suitable non-ionic detergents are the polyalkylene oxide condensates with hydrophobic organic compounds, usually of an aliphatic or an aromatic structure. Suitable examples thereof are the higher alkyl aryl polyglycol ethers (e. g. nonyl phenyl condensed with 6 to 30 moles ethylene oxide), organic acid esters with ethylene oxide (e. g. tall oil esters with 6 to 30 moles of ethylene oxide), and polyethylene glycol ethers of polyoxypropylene glycol having a molecular weight of about 2,000 to 10,000, etc.

Any suitable amount of the present novel sulfonamide may be admixed with the indicated detergents provided sufficient is used to yield the desired germicidal and bleaching properties of the composition. It is preferred that the amount of the sulfonamide be a minor amount of

4

the detergent composition. The amount of anionic or non-ionic detergent will usually be from about 5 to about 99% while the amount of sulfonamide will usually be from about .01 to 50% depending upon specific effects desired, the balance of the composition, if any, being any suitable filler, diluent, or builder materials.

These compositions may also be admixed with water-soluble inorganic builder salts, either with or without the presence of other organic detergents. Among the suitable inorganic salts are the water-soluble silicates (e. g. sodium and potassium silicate), sulfate salts (e. g. sodium sulfate), phosphate salts, preferably the polyphosphates (e. g. sodium tripolyphosphate, potassium pyrophosphate, sodium hexametaphosphate), carbonates (e. g. sodium carbonate), etc. These inorganic salts may be similarly used in any suitable proportion such as from about 5 to 99% of the composition. It is preferred to use however major amounts of these builder salts and a minor amount of the sulfonamide.

These deterative compositions may be prepared in any desired form such as toilet soap bars prepared with a soap and/or sulfate or sulfonate detergent base, in particulate form such as drum or spray-dried products, as liquids, pastes, etc.

The following examples are additionally illustrative of the compositions of the present invention, and are not to be considered as limiting the invention thereto:

Example I

A detergent composition is prepared by mixing 8 parts of sodium dodecylbenzene monosulfonmonochloramide with 92 parts of a spray-dried detergent composition containing 20% sodium dodecylbenzene sulfonate, about 40% sodium tripolyphosphate, 7% sodium silicate and the balance primarily sodium sulfate. This composition has a high degree of bleaching and germicidal power combined with a high level of detergency and foaming power, particularly in the cleansing and laundering of clothes. This composition containing the chlorinated sulfonamide has markedly enhanced stain removal properties particularly on ink, tomato and similar type stains, in comparison to a composition otherwise free of said novel sulfonamide.

Example II

A germicidal soap bar is prepared by mixing 2% of sodium dodecyl benzene monosulfonmonochloramide with soap chips comprising a mixture of 75 parts of sodium soap derived from tallow and 25 parts of sodium soap derived from coconut oil. The resulting mixture is milled and plodded to produce bars in the customary manner. The bars so produced are stable and retain their germicidal efficacy over an exceptionally long period of time.

In place of the soaps used herein, other substantially saturated soap stocks derived from vegetable or animal sources may be employed. For example, soaps derived from palm kernel oil, hydrogenated fish, lard, grease, and palm and cotton seed oils may be employed.

Example III

A powdered bleach composition is prepared as follows:

	Parts by weight
Potassium salt of keryl benzene sulfonmonochloramide	25
Pentaoxide tripolyphosphate	30
Sodium sulfate	45

This composition has foaming and deterative properties and may beneficially be used directly or in conjunction with other detergent compositions in the cleansing of soiled fabrics and other articles which are benefited by treatment with an oxidizing or bleaching agent. In place of the potassium sulfonchloramide above, effective compositions are prepared using the free sulfonchloramide and the corresponding calcium salt.

5

Example IV

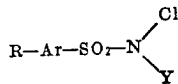
A detergent composition having desirable bleaching and germicidal properties has the following composition:

	Parts by weight	
Pentasodium tripolyphosphate-----	40	5
Tetrasodium pyrophosphate-----	15	
Sodium sulfate-----	10	
Sodium salt of dodecyl benzene sulfonmonochlor- amide-----	15	10
Sodium salt of dodecyl benzene sulfonic acid-----	20	

Although the present invention has been described with reference to particular embodiments and examples, it will be apparent to those skilled in the art that variations and modifications can be substituted therefor without departing from the principles and true spirit of the invention.

Having thus described the invention, what is claimed is:

1. A germicidal detergent composition consisting essentially of from about 5 to about 95% of a water-soluble organic anionic detergent, and from about 0.01 to 50% of a sulfonamide compound having the formula:



25

6

wherein R is an alkyl group of about 10 to 15 carbon atoms, Ar is a benzene nucleus, and Y is selected from the group consisting of chlorine, hydrogen, alkali metal, and alkaline earth metal.

2. A composition in accordance with claim 1 wherein said sulfonamide is sodium salt of dodecyl benzene monosulfonmonochloramide.

References Cited in the file of this patent

UNITED STATES PATENTS

1,699,888	Gunzler	Jan. 22, 1929
2,495,489	Van Andel	Jan. 24, 1950
2,658,916	Krems	Nov. 10, 1953

FOREIGN PATENTS

775,247	France	Oct. 8, 1934
802,506	France	June 13, 1936