3,304,263

PHOSPHINE OXIDE DETERGENT COMPOSITION John Thomas Yoke III, Tucson, Ariz., and Robert G. Laughlin, Springfield Township, Hamilton County, Ohio, assignors to The Procter & Gamble Company, Cincinnati, Ohio, a corporation of Ohio

No Drawing. Original application Feb. 16, 1962, Ser. No. 173,834. Divided and this application Sept. 18, 1963,

Ser. No. 309,841

9 Claims. (Cl. 252-132)

This application is a division of copending application 10 Serial No. 173,834 of February 16, 1962, which is a continuation-in-part of application Serial No. 96,383, filed March 17, 1961, and now abandoned.

This invention relates to novel tertiary phosphine oxide detergents and detergent compositions containing them. 15

In the constant improvement of organic detergent compounds, certain features have been found to be highly desirable. These features include resistance toward the ingredients imparting hardness to water, a high degree of detergency, and capacity for solubilization of hard water 20 soaps, such as calcium soap. Although there are a number of organic detergents which have these characteristics. detergent compounds having additional desirable characteristics find a wider scope of application.

Thermal stability is a highly desirable property which 25 is lacking in many detergents. Such stability is particularly desirable when detergents are subjected to heat during use or processing, as for example, in spray drying granular

detergent compositions.

A degree of bacteriostatic activity is also desirable in 30 organic detergent compounds.

Resistance to hydrolysis under ordinary conditions of use is also desirable.

Another advantageous property for an organic detergent is a low degree of hygroscopicity which results in improved crystallinity. When detergents which are hygroscopic are used in bar or granular form, desirable physical properties are impaired. Bars become soft and slimy and granules tend to cake and lose their free flowing and quick dissolving characteristics.

A high degree of detergency in cool or cold water is also highly desirable. Many fabrics such as those containing crease resistant additives should be washed in cool water to retain their crease resistant properties. garments should be washed in cool water to avoid shrinkage. In some locations warm or hot water is not available.

It is a principal object of this invention to provide organic detergents and detergent compositions which have excellent detergency and alkaline earth soap solubilization characteristics as well as resistance to hydrolysis, and a degree of bacteriostatic activity.

It is another object to provide preferred detergents and compositions which have these characteristics and also have high thermal stability and, in the case of the most preferred detergents and compositions, a low degree of hygroscopicity.

It is another important object to provide preferred detergents and detergent compositions which have these characteristics and also have a high degree of detergency in cool water.

It was found that these and other objects are achieved in a novel class of tertiary phosphine oxides having the structure set forth below and in detergent compositions containing such compounds as hereinafter more fully described.

The phosphine oxides of this invention are probably resonance hybrids, the major canonical forms of which

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In the above formulas, R is an alkyl, alkenyl or monohydroxyalkyl radical ranging from 10 to 18 carbon atoms in chain length and R' and R" are each alkyl or monohydroxyalkyl groups containing from 1 to 3 carbon atoms. The class of compounds described above will hereinafter be referred to more simply as  $RR'R''P \rightarrow O$ .

Examples of the compounds of this invention are:

dimethyldodecylphosphine oxide, dimethyltetradecylphosphine oxide, methylethyltetradecylphosphine oxide, dimethylhexadecylphosphine oxide, dimethyloctadecylphosphine oxide, ethylpropylhexadecylphosphine oxide, diethyldodecylphosphine oxide, diethyltetradecylphosphine oxide, dipropyldodecylphosphine oxide, bis(2-hydroxyethyl)-dodecylphosphine oxide, bis(3-hydroxypropyl)-dodecylphosphine oxide, methyl-2-hydroxypropyltetradecylphosphine oxide, dimethyloleylphosphine oxide, dimethyl-2-hydroxydodecylphosphine oxide, bis(hydroxymethyl)-dodecylphosphine oxide and diethyl-1-hydroxydodecylphosphine oxide.

Tertiary phosphine oxides as a broad class of compounds are known. It was surprising to find, however, that the particular trialkyl phosphine oxides described above have highly desirable properties for use as organic detergents.

It appears that only certain tertiary phosphine oxides have the aforementioned desired characteristics; in these certain phosphine oxides, R, R' and R" must be as described above. If R is longer in chain length than 18 carbon atoms or shorter in chain length than 10 carbon atoms, desired detergency characteristics are not obtained. Likewise, if R' and R" contain more than 3 carbon atoms, desired detergency characteristics and bacteriostatic activity are not obtained.

The most preferred tertiary phosphine oxides are the C<sub>10</sub>-C<sub>18</sub> alkyldimethylphosphine oxides; particularly desirable are C<sub>12</sub>-C<sub>14</sub> alkyldimethylphosphine oxides which have good sudsing characteristics. The C<sub>10</sub>-C<sub>18</sub> alkyldimethylphosphine oxides show bacteriostatic activity against Gram positive organisms. (Some tests indicate activity also against Gram negative organisms.) phosphine oxides also exhibit the desired high degree of cool water detergency as well as the desired low degree of hygroscopicity. The  $C_{10}$ - $C_{18}$  alkyl diethyl phosphine oxides have about as good detergency characteristics as the dimethyl compounds.

The alkenyldimethylphosphine oxides do not have as desirable non-hygroscopicity characteristics as the corresponding alkyl and hydroxyalkyldimethylphosphine oxides. The alkenyl compounds, as well as the alkyl and hydroxyalkyl compounds, have a degree of thermal stability, however.

The tertiary phosphine oxides of this invention can be prepared by methods similar to the methods for preparing the tertiary phosphine oxides which are known in the Such methods are discussed in an article by K. Darrell Berlin and George B. Butler, Chemical Reviews, volume 60, pp. 243-259, June 1960. In general, tertiary phosphine oxides of this invention can be prepared by oxidizing the corresponding tertiary phosphine, for example, with hydrogen peroxide.

In tertiary phosphine oxides of this invention, R can be derived from naturally occurring fats and oils or from synthetic sources. Mixtures of phosphine oxides are very suitable wherein the R groups vary in chain length in the C<sub>10</sub> to C<sub>18</sub> range, as for example, the alkyl groups from coconut fatty alcohol (or distilled coconut fatty alcohol).

Compounds of this invention are useful per se as detergent and surface active agents. Desirably they are used with other materials to form detergent compositions, as for example, liquid, bar, flake, granular or tabletted granular compositions. Such detergent compositions can contain the tertiary phosphine oxides of this invention and a material is selected from the class consisting of anionic organic detergents, nonionic organic detergents, water-soluble inorganic alkaline builder salts, water-soluble organic alkaline sequestrant builder salts and mixtures thereof wherein the ratio of phosphine oxide to this class of material is in the range of about 4:1 to about 1:20. (Parts, ratios and percentages herein are by weight.)

Granular or flake detergent compositions preferably contain about 5% to about 50% (desirably 10% to 15 30%) of the phosphine oxides of this invention and liquid formulations contain about 4% to about 20% of such phosphine oxides. Granular or flake detergent compositions preferably contain at least an equal amount of an alkaline builder salt. Liquid formulations preferably contain from about 8% to about 40% of a water soluble alkaline builder salt, the balance of the composition being a solvent such as water, and/or other liquid vehicles as hereinafter more fully described.

When used with anionic detergents, such as a soap base, and, if desired, inert fillers, bar formulations contain about 5% to about 50% of phosphine oxide. Bar formulations can contain about 40% to about 80% of the phosphine oxides of this invention as the only detergent component, if desired, and the balance inert fillers or builders.

Water-soluble inorganic alkaline builder salts used alone or in admixture are alkali metal carbonates, borates, phosphates, polyphosphates, bicarbonates and silicates. (Ammonium or substituted ammonium salts can also be used.) Specific examples of such salts are sodium tripolyphosphate, sodium carbonate, sodium tetraborate, sodium pyrophosphate, sodium bicarbonate, potassium tripolyphosphate, sodium hexamethaphosphate, sodium sesquicarbonate, sodium mono- and diortho phosphate and potassium bicarbonate. Such inorganic builder salts enhance the detergency of the subject phosphine oxides.

Examples of organic alkaline sequestrant builder salts used alone or in admixture are alkali metal, ammonium or substituted ammonium, aminopolycarboxylates, e.g., sodium and potassium ethylenediaminetetraacetate, sodium and potassium N-(2-hydroxyethyl)-ethylenediaminetriacetates, sodium and potassium nitrilotriacetates and sodium, potassium and triethanolammonium N-(2-hydroxyethyl)-nitrilo diacetates. Mixed salts of these polycarboxylates are also suitable. The alkali metal salts of phytic acid, e.g., sodium phytate are also suitable as organic alkaline sequestrant builder salts (see U.S. Patent

Anionic organic detergents which can be used in the compositions of this invention alone or in admixture include both the soap and non-soap detergents. Examples of suitable soaps are the sodium, potassium, ammonium and alkylolammonium salts of higher fatty acids (C<sub>10</sub>-C<sub>20</sub>). Particularly useful are the sodium and potassium salts of the mixtures of fatty acids derived from coconut oil and tallow, i.e., sodium or potassium tallow and coconut soap. Examples of anionic organic nonsoap detergents are: alkyl glyceryl ether sulfonates; alkyl sulfates; alkyl monoglyceride sulfates or sulfonates; alkyl polyethenoxy ether sulfates; acyl sarcosinates; acyl esters of isethionates; acyl N-methyl taurides; alkyl benzene sulfonates; alkyl phenol polyethenoxy sulfonates. In these compounds the alkyl and acyl groups, respectively, contain 10 to 20 carbon atoms. They are used in the form of water-soluble salts, the sodium, potassium, ammonium and alkylolammonium salts, for examples. Specific examples are: sodium lauryl sulfate; potassium Nmethyl lauroyl tauride; triethanolamine dodecyl benzene

be used in the compositions of this invention alone or in admixture are: polyethylene oxide condensates of alkyl phenols wherein the alkyl group contains from 6 to 12 carbon atoms (e.g., t-octylphenol) and the ethylene oxide is present in a molar ratio of ethylene oxide to alkyl phenol in the range of 10:1 to 25:1; condensation products of ethylene oxide with the product resulting from the reaction of propylene oxide and ethylene diamine wherein the molecular weight of the condensation products ranges from 5,000 to 11,000; the condensation products of from about 5 to 30 moles of ethylene oxide with one mole of a straight or branched chain aliphatic alcohol containing from 8 to 18 carbon atoms (e.g., lauryl alcohol); C<sub>10</sub>-C<sub>18</sub> alkyl di- $(C_1-C_2$  alkyl) amine oxides (e.g., dodecyl dimethyl amine oxide).

Where the phosphine oxides of this invention, particularly the C<sub>12</sub>-C<sub>14</sub> alkyldimethylphosphine oxides, are combined with the water soluble inorganic alkaline builder salts or organic alkaline sequestrant builder salts as described above, heavy-duty laundering compositions are obtained which have outstanding effectiveness in both cool water and hot water detergency. Preferably the ratio of phosphine oxide to builder in such compositions is in the range of 1:1 to 1:10. It is quite surprising that the phosphine oxides of this invention, when employed in heavyduty laundering compositions have outstanding cool water detergency effectiveness. These phosphine oxides are much more effective in this respect than soap, such as coconut oil soap, and are also much more effective than 30 commonly used laundering active detergents such as alkylbenzenesulfonate (the alkyl being derived from tetrapropylene) and C<sub>12</sub>-C<sub>18</sub> alkyl sulfates.

The detergent compositions of this invention can contain any of the usual adjuvants, diluents and additives, for 35 example, ampholytic, cationic or zwitterionic detergents, perfumes, anti-tarnishing agents, anti-redeposition agents, bacteriostatic agents, dyes, fluorescers, suds builders, suds depressors, and the like without detracting from the advantageous properties of the composition.

The following are examples which illustrate the tertiary phosphine oxide compounds and compositions of this invention.

## EXAMPLE I

274 grams dodecyl bromide (1.1 moles) in 1100 ml. of ether (diethyl ether) was added to 24.3 grams of magnesium turnings (1.0 mole) in 1100 ml. of ether. Addition time was 70 minutes. The resulting dodecyl magnesium bromide (Grignard reagent) in ether was cooled in ice. 100.8 grams of cadmium chloride (0.55 mole) was added to this reagent. The resulting mixture was then stirred vigorously for two hours. To this mixture was added 171.8 grams of PCl<sub>3</sub> (1.25 moles) in 1000 ml. of ether. During this addition the mixture was stirred vigorously; the addition time was 60 minutes. The temperature was kept at  $-50^{\circ}$  C. The resulting mixture was stirred an additional 60 minutes at this temperature and permitted to stand for 16 hours. This mixture was filtered and the precipitate was washed with 800 ml. of ether. The precipitate was discarded. The ether was removed from the filtrate by distillation. The residue was twice distilled; the fraction having a boiling point in the range of 135°-140° C. (at 1.5 mm. Hg) contained predominantly dichlorododecyl phosphine.

73.8 grams of the dichlorododecyl phosphine distillate (.272 mole) in 300 ml. of ether was then reacted with a Grignard reagent prepared from 84.0 grams of methyl iodide and 14.6 grams of magnesium metal in 1200 ml. of ether. The reaction was conducted in an ice-salt bath at 5° C. The Grignard reagent was added to the dichlorododecyl phosphine-ether mixture dropwise over a period of 60 minutes. The reaction mixture was stirred one hour after the addition. 140 grams of NH<sub>4</sub>Cl in 500 ml. of water was added to this mixture cautiously. 110 grams The examples of nonionic organic detergents which can 75 of Na<sub>2</sub>SO<sub>4</sub> in 600 ml. of water was then added to salt out

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the desired product to the ether layer. The layers were separated; the water layer was discarded. The ether layer was washed twice with 200 ml. of  $20\%~Na_2SO_4$  solution. The ether layer was dried over a  $CasO_4$  desiccant. The ether was removed by distillation. The residue was then 5 distilled through a micro Claisen apparatus. The weight of resulting dimethyldodecylphosphine was 26.7 grams (a yield of 43%).

24.2 grams of this dimethyldodecylphosphine (.105 mole) was stirred at room temperature with 500 ml. of water and 135 ml. of 3% H<sub>2</sub>O<sub>2</sub> (.119 mole). The temperature rose to 44° C. The reaction product was then freeze-dried. 23.5 grams of dimethyldodecylphosphine oxide was obtained (91% yield).

Elemental analysis of the resulting material showed the 15 following—

Calculated: 68.5% C, 12.6% H, 12.6% P. Found: 68.1% C, 12.5% H, 12.6% P.

The process of Example I can be essentially summarized in the following equations:

(1)  $RBr+Mg\rightarrow RMgBr$ 

(2)  $2RMgBr+CdCl_2\rightarrow (R)_2Cd+MgCl_2+MgBr_2$ 

(3)  $(R)_2Cd+2PCl_3\rightarrow 2RPCl_2+CdCl_2$ 

(4)  $RPCl_2+R'MgI+R''MgI\rightarrow RR'R''P+MgCl_2+MgI_2$ 

(5)  $RR'R''P+H_2O_2\rightarrow RR'R''P\rightarrow O+H_2O$ R, R' and R'' are as noted above.

(These reactions are not quantitative, however, and there are some side reactions.)

In the reactions outlined in the equations above in 30 Example I and the reactions outlined in the equations in Example II, the desired alkyl radicals in RR'R''P>O can be obtained by selecting the appropriate reaction products. For example, if a methylpropyldodecylphosphine oxide is desired, one mole each of methyl magnesium 35 iodide and propyl magnesium iodide are used in Equation 4 above; however, in such a reaction, a gross mixture of phosphine oxides including this compound would result.

Water solubility of dimethyldodecylphosphine oxide at 27° C. was 50 to 55%. The thermal stability of this compound is excellent, having been recovered substantially unchanged after being heated at 175° C. for two hours in a nitrogen atmosphere. This compound is very resistant to hydrolysis, breaking down only with vigorous treatment such as fusion with caustic or high temperature acidification. The low degree of hygroscopicity was shown when dimethyldodecylphosphine oxide, a crystalline solid, was allowed to stand in room air for several days without losing is crystalline form. Many other crystalline organic detergents form syrups when subjected to the same test. Further quantitative tests have shown that the water pickup by this compound at 70° F. and 80% relative humidity is very low (0.2% of sample weight).

The bacteriostatic efficacy of dimethyldodecylphosphine oxide was determined. In an aqueous matrix, it required 55 a concentration of only 17.5 to 19 p.p.m. of this compound to arrive at the bacteriostatic breakpoint of M. aureus (gram positive). Bacteriostatic breakpoint is that concentration of material at which the multiplication of an organism is stopped. This degree of bacteriostatic efficacy is significant.

## EXAMPLE II

In 3000 cc. of heptane, 34.5 grams of sodium (1.50 moles) was reacted with 291 grams of dibutyl phosphonate ( $C_4H_9O)_2P(O)H$  (.50 mole) at a temperature of 100° C. for 2 hours with stirring. 414 grams (1.50 moles) tetradecyl bromide was added to this reaction mixture over a period of 45 minutes at the same temperature. This mixture was then stirred for 6 hours. After cooling, this mixture was washed with 1500 cc. of water and 250 cc. of 2% aqueous acetic acid, then washed twice more with 2% aqueous acetic acid. The heptane was distilled off and the mixture was then distilled.

362 grams of the resulting dibutyl tetradecylphos- 75 tallow alkyl sulfate.

phonate was hydrolyzed to tetradecylphosphonic acid with 1550 ml. of hydrobromic acid. The butyl bromide formed was removed by distillation.

192 grams of the tetradecylphosphonic acid (.69 mole) was reacted slowly with 316 grams of  $PCl_5$  (1.52 moles) over a steam bath and under a  $N_2$  stream. The reaction mixture was heated with steam for 30 minutes; then  $SO_2$  was passed through the mixture for 90 minutes. Volatile impurities were removed with a vacuum pump.

62.3 grams of the resulting tetradecylphosphonic dichloride (.2 mole) in 150 cc. of ether was reacted with 0.6 mole of methyl magnesium chloride (Grignard reagent) at -73° C. The reaction mixture was stirred for 6 hours at room temperature and was permitted to stand for 16 hours. 498 cc. of a half-saturated solution of NH<sub>4</sub>Cl was added to this mixture. Two layers formed. The ether layer was removed and the aqueous layer was extracted with ether. The ether layer and the other extract from the aqueous layer were mixed and then dried.

34 grams of dimethyltetradecylphosphine oxide were obtained (a yield of 62%). The material was distilled (B.P. 138–140° C. at 1 mm. Hg) and recrystallized from hexane once. This product had the following analysis—

Calculated: 70.00% C, 12.75% H, 11.30% P. Found: 69.8% C, 12.7% H, 11.0% P.

Diethyldodecylphosphine oxide was prepared by a similar procedure except that dodecyl bromide was used, instead of tetradecyl bromide, in a molar equivalent amount.

Dimethyldodecylphosphine oxide, dimethyloctadecyl phosphine oxide and diethyloctadecylphosphine oxide were prepared by processes similar to that described in Example II. The essential equations of this process are summarized below.

R, R' and R" are as noted above.

(The reactions are not quantitative, however, and there are some side reactions.)

To determine the detergency characteristics of the phosphine oxides of this invention, the ability of these compounds to remove lipid soil from cloth was measured. Cloth swatches were soiled with a predetermined amount of artificial lipid soil (a blend of olive oil, stearic acid, oleic acid and tallow fatty alcohol). These soiled swatches were washed in a Tergotometer using water at 140° F. and containing .10% by weight of the product being tested. (Tergotometer testing is described in "Detergency Evaluation and Testing," by J. C. Harris, Interscience Publishers, Inc. (1954) page 60.) Swatches are weighed on a dry basis before and after washing. On the basis of the percent residual lipid soil, the lower the percent, the better the detergency performance.

Using soft water .10% dimethyldodecylphosphine oxide resulted in 43.9% residual soil. In this same test, sodium dodecylbenzenesulfonate, a widely used detergent ingredient for laundry detergent compositions resulted in 52.4% residual soil. Thus, dimethyldodecylphosphine oxide is superior in detergency to dodecylbenzenesulfonate; it approaches the performance of one of the best commercially used laundry detergent ingredients, sodium tallow alkyl sulfate.

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When used in a built laundry detergent formulation (20% organic detergent, 50% sodium tripolyphosphate and 30% sodium sulfate), dimethyldodecylphosphine oxide (as the organic detergent) also gave results superior to that of dodecylbenzenesulfonate and approaching that of tallow alkyl sulfate, when compared with these compounds on the same basis in the residual lipid soil Tergotometer test using water of 7 grain hardness.

Built laundry detergent compositions containing 50% sodium tripolyphosphate, 30% sodium sulfate and 20% 10 dimethyldodecylphosphine oxide, diethyldodecylphosphine oxide, dimethyltetradecylphosphine oxide or diethyltetradecylphosphine oxide, resulted in lipid soil detergency properties (using naturally soiled cloth) superior to like formulations containing dodecylbenzenesulfonate and approaching like formulations containing tallow alkyl sulfate. The same basic formulation, but containing dimethyloctadecylphosphine oxide or diethyloctadecylphosphine oxide or diethyloctadecylphosphine oxide had satisfactory detergent characteristics but not as desirable as those compositions containing the  $C_{12}$  and  $C_{14}$  homologues.

The above described phosphine oxides were all efficient solubilizers of calcium soap. All had thermal stability comparable to that described above for dimethyldodecylphosphine oxide.

The cool water detergency of the phosphine oxides of this invention was evaluated by washing naturally soiled white dress shirts. Shirts were worn by male subjects under ordinary conditions for two normal working days. The degree to which a detergent composition containing a detergent compound to be tested cleaned the collars and cuffs of the soiled shirts, relative to the cleaning degree of a similar composition containing a standard detergent compound was considered a measure of the detergency effectiveness of the test compound.

The washing solution used in the test contained 0.03% organic detergent compound and 0.06% sodium tripolyphosphate. (No fluorescers, bleaches or antiredeposition agents were used.) The pH of the washing solution was 10 and water of 7 grains per gallon hardness was used. A conventional agitator-type washer was used. The detergent compound in the standard detergent composition was sodium alkylbenzenesulfonate (the alkyl being derived from tetrapropylene), the most commonly used organic detergent in heavy-duty laundry detergent compositions. The test detergent composition contained the detergent compound to be tested, i.e., compared with the standard composition.

Under these conditions, the detergency effectiveness of dimethyldodecylphosphine oxide in wash water of 80° F. (1) was quite superior to the detergency effectiveness of sodium-alkylbenzenesulfonate at 140° F. and of dimethyldodecylamine oxide at 80° F., and (2) was markedly superior to the detergency effectiveness of sodium tallow alcohol sulfate and sodium alkylbenzenesulfonate at 80° F.

Thus, dimethyldodecylphosphine oxide showed surprising cool water detergency. Similar results are obtained with dimethyltetradecylphosphine oxide.

Similar comparative results are obtained, if in the white shirt detergency test, an organic alkaline sequestrant builder salt, sodium ethylenediaminetetraacetate or potassium nitriloacetate, is used instead of the sodium tripolyphosphate.

The surprising cool water detergency of the phosphine oxides of this invention is best seen in aqueous solutions having a temperature in the range of about 60° F. to about 95° F. and the concentration of the active phosphine oxide compound is in the range of about 0.005% to about 0.5% and the washing solution also contains a water soluble inorganic alkaline builder salt or organic alkaline sequestrant builder salt (as hereinbefore described) in the range of about 0.01% to about 1.0%.

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The following granular detergent composition was pre-

	parea:	
	Pe	rcent
	Dimethyldodecylphosphine oxide	17.5
5	Sodium sulfate	23.0
	Sodium tripolyphosphate	50.0
	Sodium silicate	6.0
	Water	3.5

A 0.436% water solution of this composition was used to wash soiled dishes. The water was at 115° F. and 7 grain hardness. 40 seconds of mechanical agitation provided more than 1" of stable suds.

The phosphine oxides of this invention can be used in effective solid form detergent compositions having improved hygroscopicity, thermal stability and both cool and hot water detergency characteristics. They have the following formulas:

## Spray-dried granular detergent

20	Perc	ent
	Diethyldodecylphosphine oxide	10
	Sodium dodecyl benzene sulfonate (The dodecyl	10
	group being derived from tetrapropylene)	
25	Sodium tripolyphosphate	
	Sodium sulfate	30
	Granular detergent	
	Dimethyltetradecylphosphine oxide	10
	Condensation product of one mole of nonyl phenol	
30	and nine moles of ethylene oxide	10
	Sodium pyrophosphate	50
	Sodium carbonate	3
	Trisodium phosphate	3
۰.	Sodium sulfate	24
35	Milled toilet bar	
	Dimethyldodecylphosphine oxide	10
	Sodium coconut oil soap	15
	Sodium tallow soap	60
40	Moisture	15
	Milled toilet bar	
	Methylethyldodecylphosphine oxide	50
	Tallow fatty acid	
45	Moisture	15
4.0	Cornstarch	5
	Triethanolamine ethylenediamine tetraacetate	5
	Scouring cleanser	
	Silica flour	85
50	Detergent consisting of 85% trisodium phosphate and	
	15% bis(2-hydroxyethyl) 2-hydroxydodecylphos-	
	phine oxide	15

While the phosphine oxides of this invention find particularly desirable utilization in solid form detergent compositions because of their improved hygroscopicity and
thermal stability characteristics, the outstanding cool
water detergency properties of the compounds provide
basis for their advantageous use in liquid detergent compositions. Liquid compositions provide convenience in
use, particularly for measurement and dispensing operations.

Outstanding effective heavy-duty, homogeneous, liquid detergent compositions can be made with the preferred C<sub>12</sub>-C<sub>14</sub> alkyldimethylphosphine oxides. Such liquid compositions comprise from about 4% to about 20% of such preferred phosphine oxides, from about 5% to about 15% of an aryl sulfonate solubilizing agent, e.g., sodium or potassium toluene, benzene or xylene sulfonate, and from about 8% to about 40% of a water soluble inorganic alkaline builder salt, organic alkaline sequestrant builder salt or mixtures thereof as described above, the balance of the composition being water. From 1% to about 12% of an anionic or nonionic non-soap organic detergent as hereinbefore de-

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scribed, can also advantageously be included. From 1% to about 5% sodium silicate as a corrosion inhibitor and additional builder can also be advantageously included as well as minor amounts of such useful additives such as perfume, dyes and opacifiers. Preferably the builder salt is potassium pyrophosphate, potassium nitrilo triacetate or potassium ethylenediaminetetraacetate.

The following formulation is an example of a homogeneous, heavy-duty liquid laundering composition which has excellent sudsing characteristics and outstanding de- 10 tergency characteristics in both cool and warm water;

Percer	nt
Sodium dodecylbenzene sulfonate (the dodecyl be-	
ing derived from tetrapropylene)	6
Alkyldimethylphosphine oxide (the alkyl being a	
	6
	20
Potassium toluene sulfonate	8
Sodium silicate 3.	
Carboxymethylhydroxyethyl cellulose 0.	.3
Perfume0	.3
Water Balance	

A composition with equivalent characteristics can be obtained by using in the above formulation an equivalent 25 amount of potassium nitrilotriacetate instead of the potassium pyrophosphate, an equivalent amount of potassium xylene sulfonate for the toluene sulfonate, and equivalent amount of the condensation product of dodecanol and 10 moles of ethylene oxide instead of the dodecyl benzene 30 sulfonate.

What is claimed is:

- 1. A detergent composition consisting essentially of a tertiary phosphine oxide detergent compound having the formula RR'R"P→O, wherein R is selected from the group consisting of alkyl, alkenyl and monohydroxyalkyl radicals ranging in chain length from 10 to 18 carbon atoms, R' and R" are each selected from the group consisting of alkyl and monohydroxyalkyl radicals containing from 1 to 3 carbon atoms and a material selected from the group consisting of anionic organic detergents nonionic organic detergents, water-soluble inorganic alkaline builder salts, organic alkaline sequestrant builder salts and mixtures thereof, the ratio of said phosphine oxide detergent compound to said material ranging from about 4:1 to about 45 1:20.
- 2. A detergent composition consisting essentially of an alkyldimethylphosphine oxide wherein the alkyl radical ranges in chain length from 10 to 18 carbon atoms and a builder selected from the group consisting of water sol- 50 uble inorganic alkaline builder salts, organic alkaline sequestrant builder salts and mixtures thereof, the ratio of said phosphine oxide to said builder ranging from about 4:1 to about 1:20.
- 3. A detergent composition consisting essentially of  $^{55}$ an alkyldimethylphosphine oxide wherein the alkyl radical

ranges in chain length from 12 to 14 carbon atoms and a builder selected from the group consisting of water soluble inorganic alkaline builder salts, organic sequestrant builder salts and mixtures thereof, the ratio of said phosphine oxide to said builder ranging from 1:1 to 1:10.

4. The composition of claim 3 wherein the builder is

sodium tripolyphosphate.

- 5. A detergent composition consisting essentially of an alkyldiethylphosphine oxide wherein the alkyl radical ranges in chain length from 10 to 18 carbon atoms and a builder selected from the group consisting of water soluble inorganic alkaline builder salts, organic sequestrant builder salts and mixtures thereof, the ratio of said phosphine oxide to said builder ranging from about 4:1 to 15 about 1:20.
- 6. A homogeneous, heavy-duty liquid detergent composition consisting essentially of from about 4% to about 20% of an alkyl dimethyl phosphine oxide compound, wherein the alkyl radical ranges in chain length from 12 20 to 14 carbon atoms, from about 5% to about 15% of an aryl sulfonate solubilizing agent selected from the group consisting of sodium and potassium toluene, benzene and xylene sulfonates, from about 8% to about 40% of a water soluble builder selected from the group consisting of inorganic alkaline builder salts, organic alkaline sequestrant builder salts and mixtures thereof, the balance of the composition being water.
  - 7. The composition of claim 6 wherein the said solubilizing agent is potassium toluene sulfonate, the said builder is potassium pyrophosphate and the said composition contains as an additional component from about 1% to about 12% of a non-soap organic detergent selected from the group consisting of anionic and nonionic detergents.
  - 8. A toilet bar composition consisting essentially of about 5% to about 50% alkyl dimethyl phosphine oxide, wherein the alkyl radical ranges in chain length from 12 to 14 carbon atoms, in a soap base, said soap being selected from the group consisting of sodium and potassium salts of higher fatty acids.
  - 9. A detergent composition consisting essentially of a tertiary phosphine oxide having the formula RR'R"P=O wherein R is an alkyl radical containing 10 to 18 carbon atoms and  $R^{\prime}$  and  $R^{\prime\prime}$  are each alkyl groups containing 1 to 3 carbon atoms, and a material selected from the group consisting of anionic organic detergents, watersoluble inorganic alkaline builder salts and mixtures thereof, the ratio of said phosphine oxide to said material ranging from about 4:1 to about 1:20.

No references cited.

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