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LIQUID DETERGENT COMPOSITION

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This invention relates to liquid detergents of the medium-duty and heavy-duty, sudsing type. The essential active ingredients in these aqueous detergent solutions are a tertiary amine oxide surface active agent and a soluble aminopolycarboxylate salt which can complex the cations of hard water.

In recent years many people have come to prefer liquid detergents for household use. The liquid form has several adavntages over the granular form, such as: convenience in pouring, ease of measurement, freedom from dust and compactness of package. The chief problem encountered in formulating heavy-duty or built liquid products has been the difficulty of including in a homegeneous solution sufficient detergent and builder to provide the performance expected of a product intended for washing soiled clothing, and other household chores. A builder is an alkaline salt which complexes the ions of hard water and which increases the level of detergency attainable with a surface active agent. In addition to the detergent and builder, it is generally desirable to include several other ingredients, which although they are present in minor quantities, serve important functions. Examples are corrosion and tarnish inhibitors, suds builders and fluorescent dyes.

Most of the development of sudsing built liquid detergents has been along the lines of formulations comprising anionic detergents with potassium pyrophosphate as a builder. The search for means of incorporating enough of these constituents, and minor constituents such as sodium silicate, in a homogeneous liquid has led to the development of complicated formulas with large numbers of ingredients. They usually include one or more suds-builders, a hydrotrope, and an organic solvent. The hydrotrope, such as potassium toluene sulfonate or potassium benzene sulfonate, and the solvent, such as ethyl alcohol, contribute little, if any, to the performance of the formulation. Thus, the expense of their incorporation must be charged solely to achievement of higher concentrations of detergent and builder in solution. Suds builders, such as fatty alkanolamides and fatty alcohols improve the sudsing performance but increase the complexity of the formula and may require a special perfume to cover their odor. Unless purified, amide-type suds builders are often undesirably colored for a liquid product.

While sodium tripolyphosphate has been widely used in granular products, it has not been widely used in liquids because of its limited solubility and its hydrolysis in aqueous solutions. This has led to the use of the more stable and soluble potassium pyrophosphate in liquids. Even potassium pyrophosprate is not as soluble and compatible with detergents as might be desired. Pyrophosphate ions will complex, or sequester, the calcium and magnesium ions of hard water, but they do not form such tight complexes as tripolyphosphate ions. Also, calcium pyrophosphate is relatively insoluble and may precipitate when a pyrophosphate-built detergent is used in hard water. This is a disadvantage of conditions are such that the precipitate deposits in cloth and gives it a harsh feel.

The above discussed problems contribute to the difficulty of formulating a built liquid product which will give a high level of detergency when employed in a

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volume small enough to appear economical to the housewife.

It is an object of this invention to provide a simplified formula for a built liquid detergent by eliminating the need for suds-builder, solvent, and in some cases hydrotrope.

An additional object of this invention is to provide a built liquid detergent containing amine oxide as the chief surface active agent and an aminopolycarboxylate as the builder with the attendant advantages of this combination.

A further object of this invention is to provide a built liquid detergent which gives a high level of cleaning at an acceptable level of usage of product.

These and other objects are accomplished by formulas containing from about 2% to about 20% of particular amine oxides and from about 10% to about 45% of soluble aminopolycarboxylate salts in aqueous solution, the total amine oxide plus aminopolycarboxylate salt level being less than about 55%.

In the following more complete disclosure of the invention, percentages refer to weight percent of the total formula unless otherwise indicated. It will be appreciated that in an aqueous system it is for the most part immaterial by which ionic salt a particular ion is introduced; for in the aqueous product there will be substantially complete and continuous exchange of ions.

Amine oxides are compounds of the general formula $R_1R_2R_3N \rightarrow O$. The arrow is a conventional representation of a semi-polar bond. They are generally prepared by the direct oxidation of the appropriate tertiary amine. When R_1 is a much longer chain than R_2 and R_3, amine oxides have surface activity. For the purpose of this invention R_1 is an alkyl radical of from 10 to 16 carbon atoms. Desirable surface active properties are lost if R_1 is substantially less than 10 carbon atoms, and solubility in the present formulation is too low if R_1 is greater than 16 carbon atoms. R_2 and R_3 are each selected from the group consisting of methyl and ethyl radicals. Preferably R_1 will be a dodecyl radical or a mixture of dodecyl wtih decyl, tetradecyl and hexadecyl radicals such that at least 50% of the radicals are dodecyl radicals. R_2 and R_3 are preferably methyl radicals. The amine oxide will be used at from about 2% to about 20% of the total formula. For best performance, more than about 5% amine oxide is preferred. Formulas of this invention containing amine oxide at a level of the order of about 5% produce surprisingly high suds and are good detergents. Too little amine oxide does not contribute sufficient detergent performance to the formulations and too much interferes with achieving builder and detergent in sufficient amount in a homogeneous liquid.

Soluble aminopolycarboxylate salts are highly suitable for builders in this invention because of their strong complexing action with calcium and magnesium ions of hard water. They also increase the level of detergency attainable. They are highly compatible with the amine oxides of the formulas of this invention. Suitable salts include the soluble salts of ethylendiaminetetraacetic acid, N-(2-hydroxyethyl)-ethylenediaminetriacetic acid, nitrilotriacetic acid and N-(2-hydroxyethyl)-nitrilodiacetic acid. Suitable cations of these soluble salts are alkali metal cations such as sodium and potassium and alkanol ammonium cations such as ethanolammonium, diethanolammonium and triethanolammonium. Alkali metal ions are the preferred cations of this invention; sodium and potassium ethylene diaminetetraacetate are the preferred aminopolycarboxylate salts. The aminopolycarboxylate salt will ordinarily be used at levels of from about 10% to about 45%. If the amount used is greater than about 32% a hydrotrope as hereinafter described is preferably added. A hydrotrope is non-essential in most of the compositions of this invention. However, a hydrotrope is needed to extend the area of homogeneous solution from about 32% aminopolycarboxylate salt to about 45% aminopolycarboxylate salt. The sum of amine oxide plus aminopolycarboxylate salt is desirably kept less than about 55% to insure sufficient water to dissolve such compositions.

Suitable hydrotropes are soluble salts of benzene sulfonate, xylene sulfonate and toluene sulfonate. The preferred hydrotropes are the alkali metal, potassium or sodium, toluene sulfonates. The hydrotrope salt may be added at levels of 0 to about 12%. If there is less than about 32% aminopolycarboxylate salt in the formula, the presence of hydrotrope is not necessary to achieve solution homogeniety at ordinary temperatures. If the aminopolycarboxylate salt level is greater than about 32%, enough hydrotrope salt to insure a homogeneous solution will be added; ordinarily about 5% is sufficient. The upper limit of less than 12% is set by increasing dilution of the product by a substantially inert ingredient. While a hydrotrope will not ordinarily be added to compositions containing less than 32% aminopolycarboxylate salt, hydrotrope can be added if so desired for any reason such as to produce a product which retains its homogeneity to a low temperature.

Most aminopolycarboxylate salts, like other sequestering agents, are corrosive towards aluminum. If the detergent product is to be used in aluminum containers or is to contact aluminum houseware for prolonged periods of time, especially at elevated temperatures, a corrosion inhibitor should desirably be included. Soluble silicates are highly effective inhibitors and can be added to certain formulas of this invention at levels of from about 3% to about 6%. Potassium, or preferably sodium silicates having a weight ratio of SiO_2:M_2O of from 1.0 to 2.8 will be used. "M" in this ratio refers to sodium or potassium. A sodium silicate having a ratio of SiO_2:Na_2O of about 1.6 is especially preferred for economy and effectiveness. In order to have enough water to dissolve the silicate-containing formulas, the amine oxide plus aminopolycarboxylate salt content should not exceed about 32%. The silicates have an additional useful property in that they, like the aminopolycarboxylate salts, are alkaline and tend to prevent the drop in pH which may occur when the product is used in certain hard waters.

A variety of long-chain organic compounds are also effective corrosion inhibitors for aluminum, and may be used in the formulations of this invention. Specific examples of effective inhibitors are the alkyl ethyleneglycol phosphates of Belgian Patent 558,448 and the alkyl glyceryl ether phosphates of U.S. patent application Serial No. 614,186, filed October 5, 1956, now U.S. Patent No. 2,892,796. In addition to preventing the corrosion of aluminum, these compounds inhibit the tarnishing of aluminum, German silver and several other non-ferrous metals. About 1% is usually required for tarnish inhibition and as much as 6% can profitably be employed for corrosion inhibition. Preferred alkyl ethyleneglycol phosphates have the general formula $R(OC_2H_4)_xOPO_3MN$. R is an alkyl radical containing 14 to 22 carbon atoms; x is an integer from 1 to 7; M and N are selected from the group hydrogen, sodium, potassium, ethanolammonium, diethanolammonium, and triethanolammonium. The preferred alkyl glyceryl ether phosphate is the reaction product of an alkyl glyceryl ether containing from 14 to 18 carbon atoms in the alkyl chain, with an amount of inorganic phosphating agent sufficient to supply at least one atom of phosphorus per molecule of alkyl glyceryl ether. The acid form, or soluble sodium, potassium, ethanolammonium, diethanolammonium, and triethanolammonium salts are employed.

Amine oxide is an effective detergent, but up to about 6% of a supplementary anionic detergent can be added to increase sudsing, increase total active content of the formula, or decrease cost. Examples of the anionic detergents which may be included are: alkyl benzene sulfonate, alkyl ethylene oxide ether sulfate wherein there are from 1 to 5 ethylene oxide residues per alkyl, alkyl glyceryl ether sulfonate and alkyl sulfate. In these compounds the alkyl chain will contain from 9 to 16 carbon atoms and preferably about 12 carbon atoms. The sulfated condensation product of about 4 moles of ethylene oxide with one mole of nonylphenol is also a suitable detergent for use in this invention. Mixtures of alkyl radicals of different chain lengths are preferably used since they are readily available from natural or synthetic sources, as from coconut alcohol and from the polymerization of propylene to give a mixture of tri-, tetra-, and pentapropylene. These detergents are used as soluble salts; specifically they are preferably used as sodium, potassium, ethanolammonium, diethanolammonium and triethanolammonium salts. The formulas of this invention, containing amine oxide, suds so well in the laundry and dishpan that no suds builder is required.

In a finished detergent formulation there will often be added in minor amounts materials which make the product more effective or more attractive. The following are mentioned by way of example. A soluble sodium carboxymethylcellulose may be added in amounts up to about 2% in inhibit soil redeposition. A tarnish inhibitor such as a benzotriazole or ethylenethiourea may be added in amounts up to about 2%. Fluorescers, perfume, and color while not essential in the compositions of the invention, may be added in amounts less than about 1%. An alkaline material or alkali such as sodium hydroxide or potassium hydroxide can be added in amounts up to about 2% to adjust pH in the absence of silicate.

All the constituents of these liquid detergent formulations are dissolved in water with the possible exception of certain of the minor non-essential ingredients which are at least finely and homogeneously dispersed. In order to insure sufficient water, the sum of aminopolycarboxylate salt and amine oxide is less than 55%. This sum is less than about 32% if silicate is included in the formula. If none of the auxiliary agents were added, the water content would thus be greater than about 45% in formula free of silicate (100%−55%), or greater than about 62% (100%−32%−6%) in silicate-containing formulations. However, the water content may be somewhat reduced from these figures by the amount of other auxiliary agents included in the formula. A maximum water level of about 80% is set by the need to get enough active ingredients into solution to hold the volume required per usage to an acceptable level. The preferred compositions will contain from about 40% to about 80% water, and from about 60% ot about 80% when silicate is present.

EXAMPLE I

The following compositions illustrative of this invention were prepared.

	A, percent	B, percent	C, percent
Dimethyldodecylamine oxide	7.5	19.0	9.2
Tetrapotassium ethylenediaminetetraacetate	20.0	9.9	23.0
Sodium silicate, SiO_2/Na_2O=1.6	4.9	5.0	4.6
Potassium coconut glyceryl ether sulfonate (the coconut alkyl radical is about 2%—C_{10}, 66%—C_{12}, 23%—C_{14} and 9%—C_{16}. About 23% of the material is coconut diglyceryl ether sulfonate)	2.5	-------	-------
Water	65.1	66.1	63.2

These compositione were clear, homogeneous, pourable liquids at room temperature. Product A, subjected to a variety of temperatures, remained clear and homogeneous at temperatures of from 0° C. to 42° C.

The performance of these samples was tested by comparing them under standard conditions with a conventional built liquid detergent. Products A to C removed more soil and increased the whiteness of the fabrics washed, more than did the conventional detergent tested under the same conditions.

EXAMPLE II

Additional formulas including sodium solicate as a corrosion inhibitor were prepared. They proved to be homogeneous, pourable liquids at room temperature. They exhibit excellent sudsing and detergent properties.

	A	B	C	D	E	F	G
	Percent						
Dimethylalkyl[1] amine oxide	12	13	10	10	8	8	5
Tetrapotassium ethylenediaminetetraacetate	16	13	18	12	20	16	13
Sodium silicate solids wt. ratio $SiO_2/Na_2O=1.6$	5	5	3.2	5	5	5	3.3
Water	Balance						

[1] The alkyl radical was substantially all dodecyl except in F in which the alkyl radicals were those of middle cut coconut alcohol (approximately 2%—C_{10}, 66%—C_{12}, 23%—C_{14} and 9%—C_{16}).

The following substitutions can be made in the compositions of this example without substantially affecting their character or performance: diethyldodecylamine oxide for dimethyldodecylamine oxide; and middle cut ethylmethylcoconutamine oxide for the dimethyldodecylamine oxide.

EXAMPLE III

The data below show compositions containing hydrotropes; some of them contain higher quantities of potassium ethylenediaminetetraacetate than the compositions of the previous examples which did not include hydrotrope. The listed compositions remain homogeneous below room temperature, are pourable, and are excellent detergents.

	A	B	C	D	E	F	G
	Percent						
Dimethyldodecylamine oxide	9.6	10.7	8	23	7.6	3.4	4.5
Tetrapotassiumethylenediaminetetraacetate	32	35.5	42	23	42	42.5	18
Potassium toluene sulfonate	5	10	10	10	7.6	3.4	4.5
Water	Balance						

Formula G also contained 5.0% sodium silicate solids having a weight ratio of SiO_2/Na_2O equal to 1.6.

Without substantially adversely affecting the appearance or performance of these compositions, potassium xylene sulfonate can be used instead of potassium toluene sulfonate.

EXAMPLE IV

The following formulas were prepared and proved to be homogeneous, pourable liquids at room temperature.

	A, percent	B, percent
Dimethyldodecylamine oxide	4.7	10
Triethanolammonium polypropylene benzene sulfonate (a mixture of tri-, tetra-, and pentapropylene averaging about C_{12})	4.6	----------
Alkali metal ethylenediaminetetraacetate[1]	25	17
Sodium silicate, wt. ratio $SiO_2/Na_2O=1.6$	4.3	5.0
Ethanol	2.8	----------
Potassium toluene sulfonate	6.0	----------
Water	Balance	Balance

[1] A—sodium and B—potassium.

Formula A gave a higher level of suds than a conventional liquid product used in a commercial washing machine with a load of soiled clothes. Each product was used at the level of 7 oz. in 12 gallons of tap water at 130° F. Formula A increased the light reflectance (whiteness) of soiled swatches more than the conventional liquid product when swatches were washed with each product under identical conditions.

Formula B also increased the whiteness of soiled swatches more than the conventional product did when they were compared under identical conditions. In use in a dishpan, copious suds were obtained initially and proved to have good stability throughout the washing of a load of soiled dishes.

EXAMPLE V

The following product was prepared and was found to be a homogeneous liquid to below 15° C. It was pourable at room temperature.

	Percent
Dimethyldodecylamine oxide	11.0
Dihydrogen tallow polyethylene glycol phosphate (the tallow radicals are those of tallow alcohol containing about 2%—C_{14}, 32%—C_{16}, and 66%—C_{18}; 4 moles of ethylene oxide were condensed with a mole of alcohol on the average)	5.9
Tetrapotassium ethylenediaminetetraacetate	40.4
Water	Balance

This composition performs well as a detergent for washing dishes or clothing; it is substantially non-corrosive towards aluminum. The sodium salt of phosphated tallow glyceryl ether may be substituted for the tallow ethylene glycol phosphate without substantially adversely affecting the performance of the product.

EXAMPLE VI

The following compositions were prepared and found to be clear homogeneous, pourable liquids at room temperature.

	A	B	C	D	E	F	G	H
	Percent							
Dimethylcoconut[1] amine oxide	9.4	6.7	12.0	9.6	12.0	8.2	10.0	10.0
Aminopolycarboxylate (as specified below)	20.3	14.1	26.0	20.8	26.0	17.8	25.0	25.0
Sodium silicate solids wt. ratio $SiO_2/Na_2O=1.6$	------	5.0	------	5.0	------	5.0	------	----
Water	70.3	74.2	62.0	64.6	62.0	69.0	65.0	65.0

[1] Alkyl radicals are those of middle cut coconut alcohol (approximately 2%—C_{10}, 66%—C_{12}, 23%—C_{14} and 9%—C_{16}).

Aminopolycarboxylate

A—tripotassium nitrilotriacetate
B—tripotassium nitrilotriacetate
C—dipotassium N-(2-hydroxyethyl)-nitrilodiacetate
D—dipotassium N-(2-hydroxyethyl)-nitrilodiacetate
E—trisodium N - (2-hydroxyethyl) - ethylenediaminetriacetate
F—trisodium N - (2-hydroxyethyl - ethylenediaminetriacetate
G—tetrasodium ethylenediaminetetraacetate
H—tetrapotassium ethylenediaminetetraacetate

These compositions give high levels of cleaning and sudsing and are well suited for general use as built liquid detergents.

What is claimed is:

1. A substantially clear homogeneous liquid detergent composition consisting essentially of: from about 2% to about 20% of an amine oxide having the general formula $R_1R_2R_3N{\rightarrow}O$, wherein R_1 contains from 10 to 16 carbon atoms with at least 50% of R_1 containing 12 carbon atoms, and wherein R_2 and R_3 are each selected from the group consisting of methyl and ethyl radicals; from about 10% to about 45% of alkali metal salt of aminopolycarboxylate selected from the group consisting of ethylenediaminetetraacetate, N-(2-hydroxyethyl)-ethylenediaminetriacetate, nitrilotriacetate, and N-(2-hydroxyethyl)-nitrilodiacetate; an amount of an alkali metal salt of a hydrotrope anion sufficient to render the liquid detergent homogeneous at room temperature, but not to

exceed 12%, selected from the group consisting of benzene sulfonate, toluene sulfonate and xylene sulfonate; and from about 40% to about 80% water; the sum of said amine oxide and said aminopolycarboxylate salt being less than about 55%.

2. The liquid detergent composition of claim 1 wherein the salt of the aminopolycarboxylate is an alkali metal ethylenediaminetetraacetate.

3. The liquid detergent composition of claim 1 wherein the salt of the aminopolycarboxylate is an alkali metal N-(2-hydroxyethyl)-ethylenediaminetriacetate.

4. The liquid detergent of claim 1 wherein the salt of the aminopolycarboxylate is an alkali metal nitrilotriacetate.

5. The liquid detergent composition of claim 1 wherein the salt of the aminopolycarboxylate is an alkali metal N-(2-hydroxyethyl)-nitrilodiacetate.

6. The liquid detergent composition of claim 1 wherein in the amine oxide R_2 and R_3 are each methyl radicals.

7. A substantially clear homogeneous liquid detergent composition consisting essentially of: from about 2% to about 20% of an amine oxide having the general formula $R_1R_2R_3N \rightarrow O$, wherein R_1 contains from 10 to 16 carbon atoms with at least 50% of R_1 containing 12 carbon atoms, and wherein R_2 and R_3 are each selected from the group consisting of methyl and ethyl radicals; from about 10% to about 45% of alkali metal salt of aminopolycarboxylate selected from the group consisting of ethylenediaminetetraacetate, N-(2-hydroxyethyl)-ethylenediaminetriacetate, nitrilotriacetate, and N-(2-hydroxyethyl)-nitrilodiacetate; an amount of an alkali metal salt of a hydrotrope anion sufficient to render the liquid detergent homogeneous at room temperature, but not to exceed 12%, selected from the group consisting of benzene sulfonate, toluene sulfonate and xylene sulfonate; as a corrosion inhibitor, from about 1% to about 6% of a soluble compound of organic phosphate anion selected from the group consisting of: $R(OC_2H_4)_xOPO_3^{\pm}$ where R is an alkyl radical containing from 14 to 22 carbon atoms and x is an integer from 1 to 7; and an anionic reaction product of an alkyl glyceryl ether containing from 14 to 18 carbon atoms in the alkyl chain, with an amount of inorganic phosphating agent sufficient to supply at least one atom of phosphorus per molecule of alkyl glyceryl ether; and from about 40% to about 80% water; the sum of said amine oxide and said aminopolycarboxylate salt being less than about 55%.

8. A substantially clear homogeneous liquid detergent composition consisting essentially of: from about 2% to about 20% of an amine oxide having the general formula $R_1R_2R_3N \rightarrow O$, wherein R_1 contains from 10 to 16 carbon atoms with at least 50% of R_1 containing 12 carbon atoms, and wherein R_2 and R_3 are each selected from the group consisting of methyl and ethyl radicals; from about 10% to about 32% of alkali metal salt of aminopolycarboxylate selected from the group consisting of ethylenediaminetetraacetate and N-(2-hydroxyethyl)-ethylenediaminetriacetate, nitrilotriacetate, and N-(2-hydroxyethyl)-nitrilodiacetate; and from about 40% to about 80% water.

9. The liquid detergent composition of claim 8 wherein the salt of the aminopolycarboxylate is an alkali metal ethylenediaminetetraacetate.

10. The liquid detergent composition of claim 8 wherein the salt of the the aminopolycarboxylate is an alkali metal N-(2-hydroxyethyl)-ethylenediaminetriacetate.

11. The liquid detergent composition of claim 8 wherein the salt of the aminopolycarboxylate is an alkali metal nitrilotriacetate.

12. The liquid detergent composition of claim 8 wherein the salt of the aminopolycarboxylate is an alkali metal N-(2-hydroxyethyl)-nitrilodiacetate.

13. The liquid detergent composition of claim 8 wherein in the amine oxide R_2 and R_3 are each methyl radicals.

14. A substantially clear homogeneous liquid detergent composition consisting essentially of: from about 2% to about 20% of an amine oxide having the general formula $R_1R_2R_3N \rightarrow O$, wherein R_1 contains from 10 to 16 carbon atoms with at least 50% of R_1 containing 12 carbon atoms, and wherein R_2 and R_3 are each selected from the group consisting of methyl and ethyl radicals; from about 10% to about 32% of alkali metal salt of aminopolycarboxylate selected from the group consisting of ethylenediaminetetraacetate, N-(2-hydroxyethyl)-ethylenediaminetriacetate, nitrilotriacetate, and N-(2-hydroxyethyl)-nitrilodiacetate; as a corrosion inhibitor from about 3% to about 6% of sodium silicate solids having a weight ratio of SiO_2 to Na_2O of from 2:1 to 1:1 and from about 60% to about 80% water and wherein the sum of said amine oxide and said aminopolycarboxylate is less than about 32%.

15. The liquid detergent composition of claim 14 wherein the salt of the aminopolycarboxylate is an alkali metal ethylenediaminetetraacetate.

16. The liquid detergent composition of claim 14 wherein the salt of the aminopolycarboxylate is an alkali metal N-(2-hydroxyethyl)-ethylenediaminetriacetate.

17. The liquid detergent composition of claim 14 wherein the salt of the aminopolycarboxylate is an alkali metal nitrilotriacetate.

18. The liquid detergent composition of claim 14 wherein the salt of the aminopolycarboxylate is an alkali metal N-(2-hydroxyethyl)-nitrilodiacetate.

19. The liquid detergent composition of claim 14 wherein the amine oxide R_2 and R_3 are each methyl radicals.

20. A homogeneous liquid detergent composition consisting essentially of: from about 5% to about 20% of an amine oxide having the general formula $R_1R_2R_3N \rightarrow O$, wherein R_1 contains from 10 to 16 carbon atoms with at least 50% of R_1 containing 12 carbon atoms, and wherein R_2 and R_3 are each methyl radicals; from about 10% to about 32% of alkali metal ethylenediaminetetraacetate; from about 3% to about 6% of sodium silicate solids wherein the weight ratio of SiO_2 to Na_2O is about 1.6; and the balance substantially water, and wherein the sum of said amine oxide and said alkali metal ethylenediaminetetraacetate is less than about 32%.

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