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DETERGENT COMPOSITIONS

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11 Claims. (Cl. 252—137)

The present invention relates to detergent compositions containing additives which impart to these detergent compositions improved sudsing characteristics at lower than usual washing temperatures and, more particularly, to improvements in the sudsing characteristics of detergent compositions containing sulfated and sulfonated anionic detergents when employed at washing temperatures as low as about 60° F.

In many areas of the world, especially Latin American countries, washing is commonly performed at temperatures below 100° F. At these low temperatures, anionic synthetic detergents have only moderate sudsing power. In addition, there is a tendency on the part of many persons when using a high-sudsing type of detergent to estimate the amount of detergent to be added to a washing solution, and particularly a dishwashing solution, on the basis of the amount of suds being formed. As a consequence, particularly when washing temperatures are below 100° F., an amount of detergent in excess of that necessary to achieve adequate detergency quite commonly will be used.

It is an object of the present invention to provide additives for anionic synthetic detergent compositions which will facilitate sudsing at temperatures below 100° F.

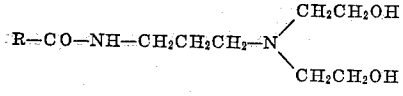
It is a further object to provide synthetic anionic sulfate and sulfonate detergent compositions which will suds at least as profusely at 60° F. as at 115° F., a conventional dishwashing temperature.

Other objects and advantageous features will appear from the following detailed description.

The present invention is based on the discovery that a limited group of amides, when present in minor amounts as components of detergent compositions, exhibit a remarkable ability to increase the sudsing capacity of the synthetic anionic sulfate and sulfonate detergents at the lower temperatures at which the more commonly employed suds-building additives are greatly reduced in efficiency.

The low temperature suds enhancing agents which are employed alone or in combination with each other in the practice of the present invention may be represented by the following structural formulae and specific compounds:

(1)

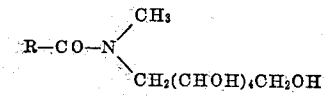


wherein R—CO is an acyl radical of a higher straight-chain fatty acid having 10–14 carbon atoms. Specific compounds are N-[3-bis (2-hydroxyethyl) aminopropyl] lauramide, N-[3-bis (2-hydroxyethyl) aminopropyl] decanamide, and N-[3-bis (2-hydroxyethyl) aminopropyl] myristamide.

These compounds can be prepared as follows: N,N-bis-(hydroxyethyl)-trimethylenediamine (1.1 moles) is added slowly, with stirring, below the surface of the fatty acid (1.0 mole) which is maintained at 165–170° F. under a

nitrogen atmosphere. After the addition is completed, the mixture is heated for an hour at 170°–180° C., then for 30 minutes under water aspirator vacuum.

(2)



wherein R—CO is an acyl radical of a higher straight chain fatty acid having 10–14 carbon atoms. Specific compounds are N-lauroyl-N-methylglucamine, N-decanoyl-N-methylglucamine, and N-myristoyl-N-methylglucamine.

These compounds can be prepared as follows: Equimolar amounts of N-methylglucamine, which is readily available commercially, and a fatty acid are heated together at 100–170° F., with constant removal of the water formed.

(3) R—NH—CO—$(CHOH)_4CH_2OH$

wherein R—NH is an amino radical and R is an alkyl radical having about 10 to about 14 carbon atoms. Specific compounds are N-keryl-D-gluconamide (the keryl group being derived from a kerosense fraction containing predominantly 12 carbon atoms per molecule), N-decyl-D-gluconamide, N-dodecyl-D-gluconamide, and N-tetradecyl-D-gluconamide.

These compounds can be prepared as follows: The del-talactone of gluconic acid, which is commercially available on large scale, is prepared by crystallization from a concentrated aqueous solution of gluconic acid. Alkyl amines having 10–14 carbon atoms are manufactured commercially. The alkyl amines are condensed rapidly and completely with glucono-delta-lactone in refluxing methanol. On cooling the solutions, high yields of the N-alkyl-D-gluconamides are obtained in the form of pure white glistening platelets which may be recrystallized from boiling ethanol or methanol.

The individual C_{10}–C_{14} fatty acids can be employed in the preparation of low temperature suds-building amides of this invention as indicated above, but mixtures of fatty acids consisting of more than 50% of such acids, such as mixed fatty acids derived from oils of the coconut group (coconut oil, palm kernel oil, etc.) can also be used. Similarly, in those instances where higher alkyl amines are employed in the preparation of the amide, individual amines or mixtures consisting of more than 50% of a C_{10}–C_{14} amine can be used.

An important feature of my invention is the fact that not all amides exhibit the quality of improving the sudsing characteristics of detergents at low temperatures. For example, the alkylolamide of monoethanolamine and the fatty acids of coconut oil, hereinafter referred to as coconut ethanolamide, is quite commonly used as an organic suds-builder; however, its maximum efficiency as a suds-builder is observed at temperatures well above 100° F. Because of the widespread use of coconut ethanolamide as a suds-builder, it has been selected as a standard for comparison with the amide suds-builders of the present invention in respect to suds-building efficacy.

Table I, for example, will serve to illustrate the stated superiority of the amides which I have found to be useful. The detergent compositions used in the comparisons expressed in Table I were homogeneous mixtures of 17.5% alkyl benzene sulfonate (the sodium salt of the sulfonic acid derived from the condensation product of benzene and polypropylenes having from 9 to 15 carbon atoms and averaging 12 carbon atoms), 47% sodium tripolyphosphate, 32.5% Na_2SO_4 and 3% of the designated amide. A standardized dishwashing test procedure was used to measure the sudsing proficiency of the detergent compositions after repeated exposure to soiled

plates, and the results are expressed as percent relative to the coconut ethanolamide composition at 115° F.

It is clearly evident from the data in Table I that N-lauroyl-N-methylglucamine, N-[3-bis (2-hydroxyethyl) amino propyl] lauramide, and N-dodecyl-D-gluconamide are outstandingly superior as suds-builders to coconut ethanolamide at a dishwashing temperature of 60° F. Furthermore, it should be noted that these three suds-building amides are at least equally as effective as coconut ethanolamide at the normal dishwashing temperature of 115° F. An interesting observation, however, that distinguishes the amides herein claimed is that they are more effective as suds-builders at 60° F. than at 115° F. On the basis of the performance of known amide builders, this is a new and unexpected property. The extraordinary efficacy of these suds-builders over a wide temperature range is without question of considerable commercial importance.

TABLE I

Product	Sudsing at 115° F., Percent	Sudsing at 60° F., Percent
Coconut ethanolamide	100	80
N-Lauroyl-N-methylglucamine	104	111
N-[3-Bis (2-hydroxyethyl) aminopropyl] lauramide	103	112
N-Dodecyl-D-gluconamide	100	102

The organic synthetic detergents which find use in the practice of this invention either singly or in admixture, can be generically defined as those possessing pronounced sudsing and detergent characteristics and having in the molecule an alkyl group of from about 8 to about 18 carbon atoms and a sulfate or sulfonate radical.

Water-soluble salts of sulfonic acids which exhibit detergent effect, such as the higher alkylated benzene sulfonic acids (e.g. potassium salt of the sulfonic acid derived from the condensation product of benzene and a chlorinated kerosene fraction containing predominantly 12 carbon atoms per molecule, or the sodium salt of the sulfonic acid derived from the condensation product of benzene and polypropylenes having from 9–15 carbon atoms and averaging about 12 carbon atoms), are greatly improved in their low temperature sudsing properties by addition thereto of a smaller amount of the amides mentioned above. Also, water-soluble salts of higher mono- fatty acid esters of 1,2-dihydroxy propane-3-sulfonic acid (sodium salt of the coconut oil fatty acid monoester of the sulfonic acid is a specific example) will find use in the practice of this invention. In addition, water-soluble salts of higher fatty acid monoesters of lower molecular weight hydroxyl alykyl sulfonic acids (e.g. oleic acid ester of the sodium salt of isethionic acid) and of the higher fatty acid amides of lower molecular amino alkyl sulfonic acids (e.g. ammonium salt of oleic acid amide of N-methyl taurine) may be employed. Likewise, detergent compositions which suds well at low temperatures may be prepared from synthetic detergents such as the water-soluble salts of the higher alcohol esters of sulfocarboxylic acids (e.g. sodium salt of the lauryl alcohol ester of sulfoacetic acid) and ethers of high molecular alcohols and lower hydroxy and polyhydroxy sulfonic acids (e.g. monolauryl ether of 1,2-dihydroxy propane-3-sodium sulfonate).

Other synthetic detergents which are useful in the practice of my invention include the water-soluble salts of high molecular aliphatic sulfuric acid esters such as the alkali metal salts of sulfuric acid esters of normal primary aliphatic alcohols having twelve to eighteen carbon atoms, particularly those whose principal active ingredient is a water-soluble salt of lauryl sulfuric acid or oleyl sulfuric acid. Specific examples are the sodium alkyl sulfate obtained from the mixed higher alcohols produced by the reduction of coconut oil, palm kernel oil, or other oils of the coconut oil group (a group of tropical nut oils characterized by their high content of combined fatty acids having ten to fourteen carbon atoms) or the sodium alkyl sulfate derived from the higher alcohols of sperm oil. Also, water-soluble salts of sulfuric acid esters of higher fatty acid monoglycerides (e.g. sodium salt of the coconut oil fatty acid monoester of 1,2-dihydroxy propane-3-sulfuric acid ester) and of the sulfated higher fatty acid alkylolamides (e.g. sodium salt of sulfated coconut fatty acid ethanolamide) may be employed. Included among these synthetic detergents, also, are the water-soluble salts of higher alkyl polyethylene oxide sulfuric acid esters (e.g. the sulfated and neutralized reaction product of about three moles of ethylene oxide with one mole of a mixture of higher alcohols derived from coconut oil and having predominantly 10–14 carbon atoms).

The above examples are merely specific illustrations of the numerous detergents which can be improved in their low temperature sudsing performance when used in conjunction with the amides heretofore designated in accordance with my invention, and is not intended that the present invention be limited thereto. These detergents may be used singly or in combination, with or without the presence of auxiliary materials which may include any of the substances employed by the art in admixture with sulfated and/or sulfonated organic synthetic detergents. The various alkali metal phosphates (e.g. tripolyphosphate and higher polyphosphates, hexametaphosphate, tetrapyrophosphate, and orthophosphate), the alkali metal silicates, sulfates, carbonates, etc. are intended to be illustrative but not preclusive examples of these auxiliary materials.

When it is desirable to incorporate an alkali metal tripolyphosphate, for example, in the detergent compositions of the present invention, any amount may be used, but if heavy-duty cleansing performance is the object, then the amount of tripolyphosphate is desirably from about 1 to 5 times the amount by weight of organic synthetic detergent salt, and preferably from 2 to 3 times.

In all such detergent compositions, the amount of amide employed to enhance the sudsing characteristics at temperatures below 100° F. will be from about 5% to about 60% by weight of the organic synthetic detergent, and preferably from about 5% to about 35%.

Thus, in the preferred compositions of the present invention having both heavy-duty cleansing performance and superior low temperature sudsing characteristics, the amount by weight of organic synthetic detergent salt will constitute from about 10% to about 40% and preferably from about 15% to about 30% of the total composition. The amide, on the same basis, will constitute from about 0.5% to about 25% and preferably from about 0.5% to about 15%, but not more than about 60% and not less than about 5% by weight of the organic synthetic detergent salt. The amount of tripolyphosphate will vary from about 1 to about 5 times the amount by weight of the organic synthetic detergent salt. It is to be understood, of course, that the respective percentages of the above three essential constituents can be adjusted to provide for the presence of sodium sulfate, which usually accompanies unpurified sulfuric reaction products, and for the inclusion of additional constituents, such as fluorescent dyes, bleaching agents, perfumes, and other constituents adapted to impart desirable properties to the composition.

The invention will be more clearly understood from the following examples, but these examples are merely illustrative of the manner in which my invention may be practiced and are not to be construed as limitations. All parts are by weight.

Example I

To 50 parts of commercial sodium alkyl benzene sulfonate containing as active ingredient about 35% of the sodium salt of the sulfonic acid derived from the condensation product of benzene and polypropylenes having

9–15 carbon atoms and averaging 12 carbon atoms are added 47 parts of sodium tripolyphosphate and 3 parts of N-lauroyl-N-methylglucamine thus giving a mixture containing 17.5% active detergent, 32.5% sodium sulfate, 47% sodium tripolyphosphate and 3% N-lauroyl-N-methylglucamine with sodium tripolyphosphate to active detergent in a ratio of 2.7:1. This mixture is superior in sudsing in the dishpan at temperatures of 60° F. to the detergent and tripolyphosphate alone and to a similar preparation containing 3% coconut ethanol amide as the suds-builder.

Example II

To 35 parts of commercial sodium alkyl sulfate (37.5% active ingredient and 62.5% sodium sulfate) prepared from higher alcohols obtained by the reduction of coconut oil and containing predominantly 10–14 carbon atoms are added 62 parts of sodium tripolyphosphate and 3 parts of N-lauroyl-N-methylglucamine. This mixture contains the sodium tripolyphosphate and active alkyl sulfate in the ratio of about 4.7 to 1 and N-lauroyl-N-methylglucamine in an amount equivalent to 3% of the total product. This mixture of sodium alkyl sulfate, sodium tripolyphosphate, sodium sulfate, and N-lauroyl-N-methylglucamine exhibits highly desirable sudsing characteristics at temperatures of 60° F., superior to those characteristics possessed by a comparable mixture containing coconut oil ethanolamide as the suds-builder.

Example III

To 50 parts of the sodium salt of alkyl glyceryl ether sulfonic acid (containing 35% active constituent) prepared from the higher alcohols obtained by the reduction of coconut oil and containing from one to three and averaging two mols of the glyceryl component per mole of alcohol used are added 47 parts of sodium tripolyphosphate and 3 parts of N-lauroyl-N-methylglucamine thus giving a mixture containing 1 part of active synthetic detergent to about 2.7 parts of sodium tripolyphosphate and N-lauroyl-N-methylglucamine in an amount equivalent to 3% of the composition. This mixture is greatly superior in sudsing proficiency at 60° F. to a comparable preparation containing 3% coconut ethanolamide instead of N-lauroyl-N-methylglucamine.

The following compositions are also productive of the desired results.

Example IV

	Percent
Sodium alkyl benzene sulfonate (the sodium salt of the sulfonic acid derived from the condensation product of benzene and polypropylenes having 9–15 carbon atoms and averaging 12 carbon atoms)	18
N-lauroyl-N-methylglucamine	1
Sodium sulfate	34
Sodium tripolyphosphate	47

Example V

	Percent
Sodium kerylbenzene sulfonate (the sodium salt of the sulfonic acid derived from the condensation product of benzene and a chlorinated kerosene fraction containing predominantly 12 carbon atoms per molecule)	10
Sodium lauryl sulfate	7.5
N-[3-bis(2-hydroxyethyl) aminopropyl] lauramide	3
Sodium sulfate	32.5
Sodium tripolyphosphate	47

Example VI

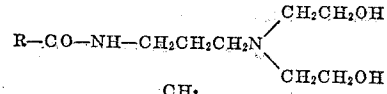
	Percent
Sodium coconut alcohol sulfates (from a fractionated mixture of coconut oil alcohols consisting predominantly of C_{10} to C_{14} fatty alcohols)	17.5
N-dodecyl-D-gluconamide	6
Sodium sulfate	32.5
Sodium tripolyphosphate	44

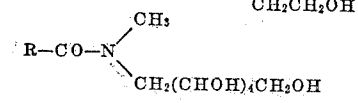
In the above examples, amides having ten or fourteen carbon atoms in the acyl or alkyl radicals may be substituted with substantially equal results.

My invention is not limited to any particular method of mixing the low temperature suds-building amides with the sulfate and sulfonate detergents with or without alkali metal tripolyphosphate. They may be incorporated in the detergent compositions in any of the forms in which these compositions are manufactured. The low temperature suds-building amides may be mechanically mixed in; may be crutched into the detergent composition in the form of a slurry; and may be dissolved in a solution of the detergent composition. While such ready-for-use mixtures may be manufactured and may be preferable for many purposes, it is likewise within the scope of my invention to add the low temperature suds-building amides to water prior to the adding of the detergent alone or detergent and alkali metal tripolyphosphate or vice versa, or to add detergent and amide or detergent, amide and alkali metal tripolyphosphate simultaneously but separately to the water.

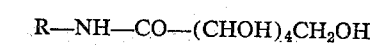
What is claimed is:

1. A cleansing and detergent composition consisting essentially of a mixture of at least one water-soluble salt of an organic sulfuric reaction product having in its molecular structure an alkyl group of from about 8 to about 18 carbon atoms and a radical selected from the group consisting of sulfonic acid and sulfuric acid, the said salt having pronounced detergent power, and at least one amide selected from the group consisting of:





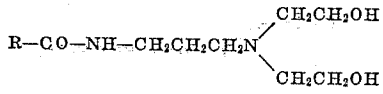
and



wherein R—CO is an acyl radical of a fatty acid having about 10 to about 14 carbon atoms and R—NH is an amino radical and R is an alkyl radical having from about 10 to about 14 carbon atoms, the amount by weight of the amide being from about 5% to about 60% of the amount by weight of said water-soluble salt and sufficient to enhance, at temperatures below 100° F., the sudsing properties of said water-soluble salt.

2. The cleansing and detergent composition of claim 1 containing an amount by weight of said amide of from about 5% to about 35% of the amount by weight of said water-soluble salt.

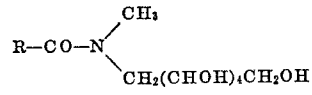
3. A cleansing and detergent composition consisting essentially of a mixture of at least one water-soluble salt of an organic sulfuric reaction product having in its molecular structure an alkyl group of from about 8 to about 18 carbon atoms and a radical selected from the group consisting of sulfonic acid and sulfuric acid, the said salt having pronounced detergent power, and an amide having the following structural formula:



wherein R—CO is an acyl radical having from about 10 to about 14 carbon atoms, the amount by weight of the amide being from about 5% to about 35% of the amount by weight of said water-soluble salt and sufficient to enhance, at temperatures below 100° F., the sudsing properties of said water-soluble salt.

4. The cleansing and detergent composition of claim 3 in which said water-soluble salt having pronounced detergent power is a mixture of water-soluble salts of alkylated benzene sulfonic acids having predominantly 9 to 15 carbon atoms in the alkyl group.

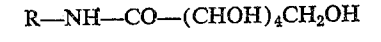
5. A cleansing and detergent composition consisting essentially of a mixture of at least one water-soluble salt of an organic sulfuric reaction product having in its molecular structure an alkyl group of from about 8 to about 18 carbon atoms and a radical selected from the group consisting of sulfonic acid and sulfuric acid, the said salt having pronounced detergent power, and an amide having the following molecular structure:



wherein R—CO is an acyl radical of a fatty acid having from about 10 to about 14 carbon atoms, the amount by weight of the amide being from about 5% to about 35% of the amount by weight of said water-soluble salt, and sufficient to enhance, at temperatures below 100° F., the sudsing properties of said water-soluble salt.

6. The cleansing and detergent composition of claim 5 in which said water-soluble salt having pronounced detergent power is a mixture of water-soluble salts of alkylated benzene sulfonic acids having predominantly 9 to 15 carbon atoms in the alkyl group.

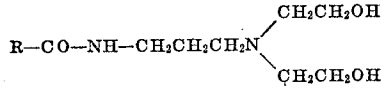
7. A cleansing and detergent composition consisting essentially of a mixture of at least one water-soluble salt of an organic sulfuric reaction product having in its molecular structure an alkyl group of from about 8 to about 18 carbon atoms and a radical selected from the group consisting of sulfonic acid and sulfuric acid, the said salt having pronounced detergent power, and an amide having the following molecular structure:

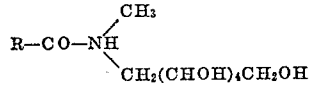


wherein R—NH is an amino radical and R is an alkyl radical having from about 10 to about 14 carbon atoms, the amount by weight of the amide being from about 5% to about 35% of the amount by weight of said water-soluble salt and sufficient to enhance, at temperatures below 100° F., the sudsing properties of said water-soluble salts.

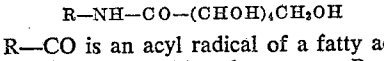
8. The cleansing and detergent composition of claim 7 in which said water-soluble salt having pronounced detergent power is a mixture of water-soluble salts of sulfated benzene sulfonic acids having from 9 to 15 carbon atoms in the alkyl group.

9. A cleansing and detergent composition consisting essentially of from about 10% to about 40% by weight, based on total product, of a mixture of water-soluble alkyl benzene sulfonate detergents, having from 9 to 15 carbon atoms in the alkyl group, an alkali metal tripolyphosphate in an amount by weight of from about 1 to about 5 times the amount by weight of said alkyl benzene sulfonate, and at least one amide selected from the group consisting of:



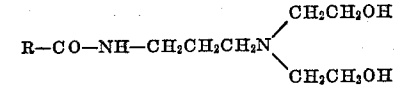


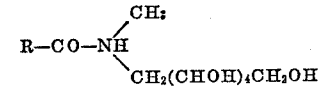
and



wherein R—CO is an acyl radical of a fatty acid having from about 10 to about 14 carbon atoms, R—NH is an amino radical and R is an alkyl radical having from about 10 to about 14 carbon atoms, the amount by weight of the amide being from about 5% to about 60% of the amount by weight of said alkyl benzene sulfonate detergent, and sufficient to enhance, at temperatures below 100° F., the sudsing properties of said alkyl benzene sulfonate detergent.

10. A cleansing and detergent composition consisting essentially of from about 15% to about 30% by weight, based on total product, of a mixture of water-soluble alkyl benzene sulfonate detergents, having from 9 to 15 carbon atoms in the alkyl group, and alkali metal tripolyphosphate in an amount by weight of from about 2 to 3 times the amount by weight of said alkyl benzene sulfonate, and at least one amide selected from the group consisting of:





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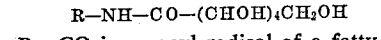
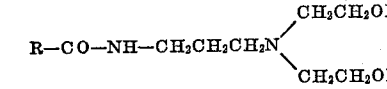
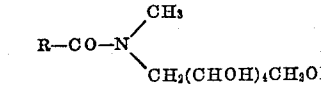


wherein R—CO is an acyl radical of a fatty acid having from about 10 to 14 carbon atoms, R—NH is an amino radical and R is an alkyl radical having from about 10 to about 14 carbon atoms, the amount by weight of the amide being from about 5% to about 35% of the amount by weight of said alkyl benzene sulfonate detergent, and sufficient to enhance, at temperatures below 100° F., the sudsing properties of said alkyl benzene sulfonate detergent.

11. A laundering composition having improved sudsing performance in aqueous solution at temperatures below 100° F., consisting essentially of (*a*) about 10% to about 40% by weight, based on total product, of a water-soluble salt of an organic sulfuric reaction product having in its molecular structure a radical selected from the group consisting of sulfonic acid and sulfuric acid ester radicals, the said salt having pronounced detergent power; (*b*) from about 0.5% to about 25%, based on the total product, but not more than 60% and not less than 5% by weight of the organic synthetic detergent salt, of at least one amide selected from the group consisting of:





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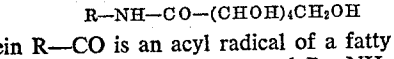

wherein R—CO is an acyl radical of a fatty acid having about 10 to 14 carbon atoms and R—NH is an amino radical and R is an alkyl radical having from about 10 to about 14 carbon atoms; and (*c*) the balance of the composition comprising alkali metal tripolyphosphate in amount from about 1 to about 5 times the amount by weight of the alkyl benzene sulfonate.

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UNITED STATES PATENT OFFICE

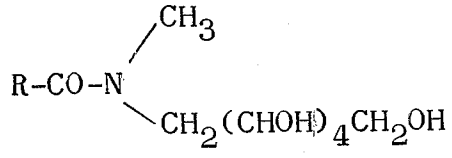
CERTIFICATION OF CORRECTION

Patent No. 2,965,576 December 20, 1960

Eugene R. Wilson

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 7, lines 60 to 63, and column 8, lines 17 to 20, the formula should appear as shown below instead of as in the patent:



column 8, line 25, after "to" insert -- about --.

Signed and sealed this 24th day of October 1961.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents