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RUST PREVENTIVE COMPOSITIONS CONTAINING MONOAMIDOCARBOXYLIC ACIDS

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16 Claims. (Cl. 252—392)

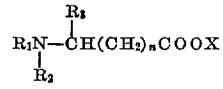
This invention relates to rust preventive oleaginous compositions. More particularly the invention relates to the use of certain nitrogenous corrosion or rust inhibitors in compositions containing predominantly an oleaginous vehicle, and which compositions may become contaminated with water or steam.

The prevention of rust formation is particularly important in the protection of metal surfaces, particularly ferrous surfaces, where such surfaces are to be lubricated in the presence of contaminating moisture. Moisture may enter lubricating systems of land and marine turbine engines, for example, by leakage through steam glands and through water-cooled heat exchangers or simply by condensation from the atmosphere. The presence of rust is injurious because it causes excessive wear of gears and bearing surfaces, and may also clog the hydraulic governor system, which last result may lead to dangerous operating speeds. Furthermore, the presence of rust has been shown to increase the rate of oxidative breakdown of the lubricant. The prevention of rusting is also essential in the moving parts of hydraulic systems generally, such as hydraulic bailing machinery, presses, etc. Other important applications of rust preventive compounds are in preservative oils, slushing oils, flushing oils, etc.

A great many materials have been proposed for the prevention of rust formation of metal surfaces, however, relatively few have been applicable in practice for the protection of lubricant systems because most are deleterious to the lubricant, the lubricant system or are disadvantageous in some other way. It has been proposed to add various carboxylic acid amides to hydrocarbon oils either for rust prevention or in order to improve the film strength and adhesion to metals. U. S. Patent No. 2,462,358 incorporates an acetoacetyl amide with an acyl residue which is of a higher fatty acid or naphthenic acid, such as $C_{17}H_{37}CONHC_2H_4NHCOCH_2COCH_3$, for the latter purpose. And U. S. Patent No. 2,403,293 discloses the incorporation of oleic monoamide of ethylene diamine or of polyethylene polyamines, purportedly a rustproofing composition. There has also been issued U. S. Patent No. 2,270,113 on esters of glycine or alanine added to oils to inhibit corrosion.

According to the present invention, it has now been found that the corrosive effect of lubricating oils and greases (oleaginous vehicle) upon bearing surfaces, gears and other metallic parts in the presence of water may be prevented by the incorporation in said oleaginous vehicle of a small amount of a class of compounds which are monoamidocarboxylic acids and their salts in which a high molecular weight aliphatic acid residue is bound to the amide nitrogen, and more specifically characterized below. Such compounds, it has been determined on a scientific basis, effectively prevent the rusting of metal surfaces, particularly those of ferrous metals, upon exposure to water or steam, when used in small amounts in lubricating compositions. These additives moreover prevent rusting without influencing the lubricant action disadvantageously.

The rust preventive compositions of this invention consist of major amounts of an oleaginous vehicle and a minor amount of the class of compounds monoamidocarboxylic acids having the empirical formula

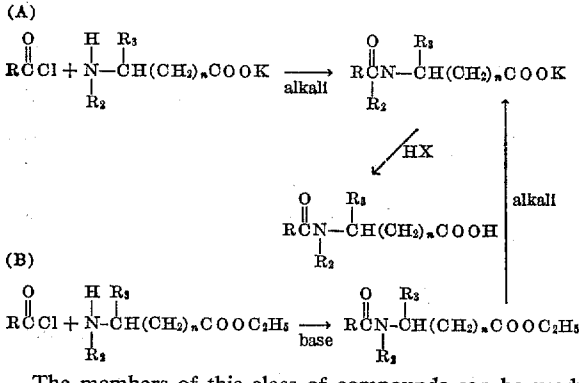


where R_1 represents an aliphatic carboxylic acid residue of C_8–C_{24}, or alkyl phosphoryl, alkyl phosphonyl, alkyl sulfonyl wherein the alkyl radical has this same number of carbon atoms; where R_2 represents an alkyl, hydroxyalkyl, phenyl group or H; R_3 represents an alkyl, carboxyalkyl groups or H; n represents 0 or 1; X represents H or groups capable of forming salts with the carboxylic acid groups such as ammonium or substituted ammonium groups, e. g. organic amines, metals, etc.

The higher molecular weight acid in the amide group can be any of the higher fatty acids, either saturated or olefinic unsaturated, of this chain length, such as from caprylic acid through behenic in the saturated series. Many of these higher saturated and unsaturated fatty acids occur as glycerides in fats and oils in nature or as esters of monohydric alcohols in waxes. But higher molecular weight aliphatic acids within this general class which do not occur naturally, such as the mixed carboxylic acids in oxidized liquid or solid hydrocarbons, can be used. It will be understood that mixed acids can be used of any of these sources, i. e., mixed higher fatty acids, or the synthetic mixed aliphatic acids from hydrocarbon oxidation, or mixtures of each, etc.

The members of this class of compounds, which are the additives of the present invention, may be regarded as derivatives of the natural and synthetic amino acids, for example derivatives of glycine, α or β alanine, sarcosine, aspartic acid, glutamic acid, leucine and other better known amino acids. These amidocarboxylic acids and their salts can be prepared in a number of ways, e. g. as outlined in Equations A or B.

(A)



(B)

The members of this class of compounds can be used alone in an oleaginous vehicle or -solvent. The latter may be termed the lubricating oil or -grease when the designation is thought of broadly. When used in an oleaginous vehicle, these compounds should be soluble, miscible or dispersable in the vehicle. If the amidocarboxylic acid or its salts is not soluble therein, at least in the percentage added solubilizers may be added which bring this about; a suitable agent for this purpose is disclosed subsequently. The term "oleaginous vehicle" as used in the specification and claims includes mineral lubricating oils derived from the refinement of petroleum or any of the so-called non-mineral oils, such as animal, vegetable oils, fats, the synthetic polyesters or organic acids, polysiloxane, polyalkylene glycols, polyolefins, also the rust-proof bases which may be used as the medium of application of rust preventive additive to the metal surface.

Some members of the lubricating oil base will be preferred for specific applications while other members will be preferred for other technical, industrial applications, where rust prevention is the objective. For it is obvious that the type of lubricating oil or grease for such surfaces as machine parts, piston rings, machine guns, light arms, gears, turbines, and the moving parts of hydraulic baling machinery, or presses, metal drums, etc., will vary greatly.

Some members of this class of compounds, namely the monoamidocarboxylic acid additives, will be preferred for specific industrial applications, while other members will be preferred for other technical applications where rust prevention is desired. Some members of this class of compounds are particularly applicable for turbine lubrication, because lubricant compositions containing them permit ready separation of entrained water. It is essential that water be easily separable from turbine lubricants by auxiliary oil purification equipment such as settling tanks, filters, blotter presses, centrifuges, etc., because water, in addition to causing rusting, accelerates oil breakdown, causes excessive foaming and facilitates the dispersion of other contaminants which accelerate oil breakdown and are injurious to the lubricating system in other ways.

These aims are achieved in practice by specifying that the turbine oil lubricant pass certain rust preventive and demulsibility tests. One of the rust preventive tests which has been widely used is the "Static Water Drop Corrosion Test" developed by Zisman et al. and described in "Industrial and Engineering Chemistry," volume 41, page 137 ff. (1949). Briefly, this test involves observing rusting at 140° F. in the presence of a drop of distilled water in the dimple of a triangular cold-rolled steel specimen immersed in the test oil. An adequately rust-inhibited oil will prevent rusting for several days while a straight mineral oil will permit rusting within several hours. The demulsibility properties of lubricating oils are characterized by the Federal Standard Stock Catalog method 320.1.5 dated November 15, 1948, and entitled "Emulsion (Lubricating Oils)." Briefly, this test involves mixing 40 milliliters of water and 40 milliliters of test oil at 130° F. or 180° F. under standard conditions and observing the separation of oil and water phases at the test temperature. The test temperature selected is usually 130° F. for light viscosity turbine oils up to about 500 Saybolt Universal seconds at 100° F. The U. S. Navy specifies that 2190–T grade turbine oils separate from water in this test within 30 minutes and have not more than 3 milliliters of an intermediate oil and water phase at this time in order to comply with Navy 14–0–15 Specifications. This criterion of demulsibility is widely adopted by turbine oil producers and consumers.

The amount of corrosion inhibitor incorporated in the finished lubricating composition may be very small, amounts of the order of 0.0125%–1%, based on the total finished oil lubricant, being sufficient to secure metallic corrosion prevention, more particularly where the surface is ferrous in nature. Greater amounts may be added as dictated by particular requirements and economic consideration.

Some of the more important uses of the various types of amido mono-carboxylic acids of the present invention, aside from turbine lubrication, are the following: Metal drawing and forming operations, gears and bearings under conditions of extreme pressure, motor oil for internal combustion engines, and top cylinder lubricants.

The following examples are offered to illustrate, but not to limit, the manner in which the present invention may be practised.

EXAMPLE I

7.5 grams of glycine was dissolved in 100 milliliters of water containing 4.0 grams of sodium hydroxide. 30.2 grams of stearoyl chloride and 50 milliliters of aqueous sodium hydroxide containing 4.0 grams of sodium hydroxide were simultaneously added to the alkaline glycine solution over a period of 20 minutes while maintaining the temperature of the mixture at about 25 to 35° C. The reaction mixture became turbid and viscous and was stirred for an additional hour. It was then neutralized to pH 2 with dilute hydrochloric acid. The precipitate which formed was filtered, washed with water and air-dried for two days. It weighed 31.6 grams. Recrystallization from ethyl acetate yielded a white product which melted at 123° C. and had a neutralization equivalent weight of 347. The theoretical neutralization equivalent weight of (N-stearoyl) glycine is 341.

The product described in the example was tested for its rust preventive properties by dissolving it in a solvent-refined and filtered non-additive turbine grade lubricating mineral oil of 150 Saybolt Universal seconds viscosity at 100° F. and subsequently testing this oil solution by the "Static Water Drop Corrosion Test" described above.

EXAMPLE II

10.2 grams of 86 percent sarcosine and 6.1 grams of magnesium oxide were dispersed in 200 milliliters of water and 30.2 grams stearoyl chloride was added dropwise over a period of thirty-five minutes. The reaction mixture was stirred for an additional 1.5 hours at room temperature and then made acid to pH 2 with dilute hydrochloric acid. The precipitate which formed was filtered, washed with water and air-dried. The yield was 32 grams of a tan solid having a neutralization equivalent weight of 364. The theoretical neutralization equivalent weight for (N-stearoyl) sarcosine is 355.5. After recrystallization from n-hexane, the purified material melted at 65 to 66.5° C.

The product described in the example are tested for its rust preventive properties by dissolving it in a solvent-refined and filtered non-additive turbine grade lubricating mineral oil of 150 Saybolt Universal seconds viscosity at 100° F. and subsequently testing this oil solution by the "Static Water Drop Corrosion Test" and "Emulsion Test" described above.

EXAMPLE III

11.8 grams of 75.5 percent sodium sarcosine was dissolved in 100 milliliters of water and 21.7 grams of lauryl chloride and 54 grams of aqueous sodium hydroxide containing 4.0 grams of sodium hydroxide were added dropwise simultaneously over a period of 30 minutes. On acidification of the mixture to pH 2 with 6 N hydrochloric acid a buttery white solid separated which was extracted with ether; the ethereal solution was thereupon dried over anhydrous sodium sulfate. On removal of ether, the residue weighed 21.3 grams and had a neutralization equivalent weight of 275. The theoretical neutralization equivalent weight for (N-lauroyl) sarcosine is 271. After crystallization from n-hexane (N-lauroyl) sarcosine had a melting point of 45 to 47° C.

The product described in the example was tested for its rust preventive properties by dissolving it in a solvent-refined and filtered non-additive turbine grade lubricating mineral oil of 150 Saybolt Universal seconds viscosity at 100° F. (37.8° C.) and subsequently testing this oil solution by the "Static Water Drop Corrosion Test" described above.

EXAMPLE IV

5.4 grams of α alanine was dissolved in 62.7 milliliters of 1.00 N NaOH solution 18.5 grams of stearoyl chloride and 65 milliliters of 1.00 N aqueous caustic soda were added dropwise simultaneously over a period of 25 minutes. The reaction mixture was stirred for an additional 30 minutes and then acidified with 6 N hydrochloric acid to pH 2. The precipitate which formed was filtered, washed with water and allowed to air-dry, yielding 21.3 grams of a white solid. After recrystallization successively from acetone and carbon tetrachloride, the isolated (N-stearoyl) α alanine melted at 108–110° C. and had a neutralization equivalent weight of 360. The

theoretical neutralization equivalent weight of (N-stearoyl) α alanine is 356.

The product described in the example was tested for its rust preventive properties by dissolving it in a solvent-refined and filtered non-additive turbine grade lubricating mineral oil of 150 Saybolt Universal seconds viscosity at 100° F. and subsequently testing this oil solution by the "Static Water Drop Corrosion Test" and "Emulsion Test" described above.

EXAMPLE V

8.9 grams of β alanine was dissolved in 100 milliliters of water containing 4.0 grams of sodium hydroxide. 30.2 grams of stearoyl chloride and 25 milliliters of aqueous sodium hydroxide containing 4.0 grams of sodium hydroxide were added dropwise simultaneously over a period of thirty minutes. The reaction mixture was stirred for an additional hour at room temperature and then made acid to pH 2 with dilute hydrochloric acid. The precipitate which formed was filtered, washed and air-dried, but since it showed a considerable ash on ignition when tested, it was redissolved in 500 milliliters of aqueous alkali containing 200 milliliters of 1.0 N sodium hydroxide in which it was clear when hot. This solution was also cooled to room temperature, then made acid to pH 2 as had been done in a prior step. The precipitate which formed was filtered, washed with water and air-dried. The dried precipitate weighed 36.0 grams and had a neutralization equivalent weight of 368. The theoretical neutralization equivalent weight of (N-stearoyl) β alanine is 356. On recrystallization from ethyl acetate the purified (N-stearoyl) β alanine melted at 119–120° C.

The product described in the example was tested for its rust preventive properties by dissolving it in a solvent-refined and filtered non-additive turbine grade lubricating mineral oil of 150 Saybolt Universal seconds viscosity at 100° F. and subsequently testing these oil solutions by the "Static Water Drop Corrosion Test" described above.

Solubilization of some of these amidomonocarboxylic acids synthesized in the above examples was effected when required by the addition of a solubilization agent which per se in oil solution had little if any rust preventive properties. The agent used was a commercially available primary, aliphatic amine, which has the tertiary-alkylamine structure, the tertiary alkyl groups having from 18–24 carbon atoms. Emulsion tests were also run on some of these oil blends. The test results are given in Table I.

It is evident that the rust preventive effectiveness of lubricating oil solutions of the monoamidocarboxylic acids is much improved over the base oil alone or with the solubilizer in it. It is also evident that the monoamidocarboxylic acids are qualitatively superior in rust preventive effectiveness than a typical high molecular weight fatty acid, such as stearic acid or the latter admixed with this identical solubilizer.

While the utility of our invention is illustrated by reference to its adaptability to turbine grade mineral lubricating oils, it is to be understood that this is intended to be merely illustrative and not a limitation of the scope thereof. Thus our invention is applicable to other oils, whether or not comparable in specification and origin, including emulsifiable soluble oils, liquid fuels, greases, and in general compositions in connection with which the water-corrosion of the containing system is to be avoided.

Having disclosed the nature of our invention and the manner in which it may be practiced, what we claim and desire to protect by Letters Patent are the following:

1. A rust preventive oleaginous composition adapted for use in the presence of water, in systems containing metal susceptible of corrosion by water, comprising an

Table I.—Rust preventive and demulsibility properties of aminocarboxylic acids in lubricating oil

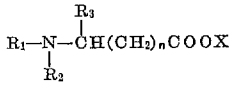
Additive (A)	Example No. (A)	Wt. Percent (A)	Wt. Percent Primene JMR [1]	Emulsion Test	"Static Water Drop Corrosion Test" Time to Rust (Hours)
Base Oil		None	None	Pass	Less than 16.
Stearic Acid		0.20	None	Pass	Do.
"Primene JMR" [1]		None	0.20	Pass	Do.
Stearic Acid and "Primene JMR."		0.20	0.20		Less than 2.
(N-stearoyl) glycine	I	0.025	0.025		More than 200.
(N-stearoyl) sarcosine	II	0.0125	None	Pass	Do.
(N-lauroyl) sarcosine	III	0.0125	0.0125		Do.
(N-stearoyl) α alanine	IV	0.0125	0.0125	Pass	Do.
Do	IV	0.20	None		Do.
(N-stearoyl) β alanine	V	0.10	0.10		Do.

[1] "Primene JMR" is a mixture of highly branched, aliphatic primary amines having the tertiary-alkylamine structure in which the primary amino nitrogen is directly attached to a tertiary carbon atom; it is composed principally of amines from eighteen to twenty-four carbon atoms and the predominant portion may be represented by the formula, $t-C_{18-24}H_{37-49}NH_2$. It contains about 8–10% non-amine material. It is a non-viscous liquid, colorless to straw colored, insoluble in water and does not dissolve water to an appreciably extent. It exhibits solubility in hydrocarbon solvents and glyceride oils or fats, and in other organic liquids previously referred to under the term "oleaginous vehicles." Additional physical properties are:

Formula	Principally $t-C_{18}H_{37}NH_2$ to $t-C_{24}H_{49}NH_2$.
Molecular Weight	Principally 269–353.
Specific Gravity, 25° C.	0.828.
Refractive Index, 25° C.	1.455.
Boiling Point (or Range)	5–95% at 274–340 C. (765 mm.).
Neutralization Equivalent	301.
Flash Point (Tag., open cup)	250° F.
Color, Varnish Scale	4—.

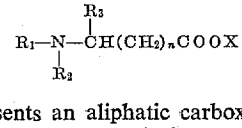
This product designated by the trademark "Primene JMR" is a product of Rohn & Hass Company.

oleaginous vehicle and a monoamidocarboxylic acid having the following empirical formula:



where R_1 represents an aliphatic carboxylic acid residue of C_8–C_{24}, R_2 represents a substituent selected from the group consisting of H, alkyl, hydroxylalkyl and phenyl group, R_3 represents a substituent selected from the group consisting of H, alkyl and carboxyalkyl, and n represents 0 or 1, and X represents H and groups capable of forming salts with the carboxylic acid group, said monoamidocarboxylic acid being present in said oleaginous composition in minor amounts, as little as a fraction of 1% to amounts greater than 1% but in sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check.

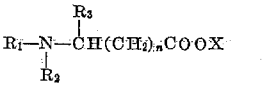
2. A rust preventive oil composition adapted for use in the presence of water, in systems containing metal susceptible of corrosion by water, comprising a hydrocarbon oil and a monoamidocarboxylic acid having the following empirical formula:



where R_1 represents an aliphatic carboxylic acid residue of C_8–C_{24}, R_2 represents a substituent selected from the group consisting of H, alkyl, hydroxylalkyl and phenyl group, R_3 represents a substituent selected from the group consisting of H, alkyl and carboxyalkyl, and n represents 0 or 1, and X represents H and groups capable of forming salts with the carboxylic acid group, said monoamidocarboxylic acid being present in said oil composition in minor amounts, as little as a fraction of 1% to amounts greater than 1% but in sufficient quantity to

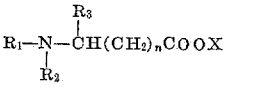
inhibit the corrosion of said metal to a great degree when compared with the check.

3. A rust preventive oleaginous composition adapted for use in the presence of water, in systems containing metal susceptible of corrosion by water, comprising an oleaginous vehicle and a monoamidocarboxylic acid having the following empirical formula:



where R_1 represents an aliphatic carboxylic acid residue of C_8–C_{24}, R_2 represents a substituent selected from the group consisting of H, alkyl, hydroxylalkyl and phenyl group, R_3 represents a substituent selected from the group consisting of H, alkyl and carboxyalkyl, and n represents 0 or 1, and X represents H and groups capable of forming salts with the carboxylic acid group, said monoamidocarboxylic acid being present in said oleaginous composition in minor amounts, as little as a fraction of 1% to amounts greater than 1% but in sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check, and an alkyl primary amine having 18–24 carbon atoms in sufficient amount to solubilize said monoamidocarboxylic acid.

4. A rust preventive oil composition adapted for use in the presence of water, in systems containing metal susceptible of corrosion by water, comprising a hydrocarbon oil and a monoamidocarboxylic acid having the following empirical formula:



where R_1 represents an aliphatic carboxylic acid residue of C_8–C_{24}, R_2 represents a substituent selected from the group consisting of H, alkyl, hydroxylalkyl and phenyl group, R_3 represents a substituent selected from the group consisting of H, alkyl and carboxyalkyl, and n represents 0 or 1, and X represents H and groups capable of forming salts with the carboxylic acid group, said monoamidocarboxylic acid being present in said oil composition in minor amounts, as little as a fraction of 1% to amounts greater than 1% but in sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check, and an alkyl primary amine having 18–24 carbon atoms in sufficient amount to solubilize said monoamidocarboxylic acid.

5. A rust-preventive composition adapted for use in the presence of water, in systems containing metal susceptible of corrosion by said water, comprising an oleaginous vehicle and minor amounts of (N-stearoyl) glycine of as little as a fraction of 1% to amounts greater than 1% but in a sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check.

6. A rust-preventive composition adapted for use in the presence of water, in systems containing metal susceptible of corrosion by said water, comprising an oleaginous vehicle and minor amounts of (N-stearoyl) sarcosine of as little as a fraction of 1% to amounts greater than 1% but in sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check.

7. A rust-preventive composition adapted for use in the presence of water, in systems containing metal susceptible of corrosion by said water, comprising an oleaginous vehicle and minor amounts of (N-lauroyl) sarcosine of as little as a fraction of 1% to amounts greater than 1% but in sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check.

8. A rust-preventive composition adapted for use in the presence of water, in systems containing metal susceptible of corrosion by said water, comprising an oleaginous vehicle and minor amounts of (N-stearoyl) alanine of as little as a fraction of 1% to amounts greater than 1% but in sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check.

9. A rust-preventive composition adapted for use in the presence of water, in systems containing metal susceptible of corrosion by said water, comprising a mineral oil and minor amounts of (N-stearoyl) glycine of as little as a fraction of 1% to amounts greater than 1% but in sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check.

10. A rust-preventive composition adapted for use in the presence of water, in systems containing metal susceptible of corrosion by said water, comprising a mineral oil and minor amounts of (N-stearoyl) sarcosine of as little as a fraction of 1% to amounts greater than 1% but in sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check.

11. A rust-preventive composition adapted for use in the presence of water, in systems containing metal susceptible of corrosion by said water, comprising a mineral oil and minor amounts of (N-lauroyl) sarcosine of as little as a fraction of 1% to amounts greater than 1% but in sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check.

12. A rust-preventive composition adapted for use in the presence of water, in systems containing metal susceptible of corrosion by said water, comprising a mineral oil and minor amounts of (N-stearoyl) alanine of as little as a fraction of 1% to amounts greater than 1% but in sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check.

13. A rust-preventive composition adapted to use in the presence of water, in systems containing metal susceptible of corrosion by said water, comprising a mineral oil and minor amounts of (N-stearoyl) glycine, as little as a fraction of 1% but in a sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check, and alkyl primary amines having 18–24 carbon atoms in sufficient amount to solubilize said (N-stearoyl) glycine, in the mineral oil, the said alkyl amines being soluble in the mineral oil but insoluble in water.

14. A rust-preventive composition adapted to use in the presence of water, in systems containing metal susceptible of corrosion by said water, comprising a mineral oil and minor amounts of (N-stearoyl) sarcosine, as little as a fraction of 1% but in a sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check, and alkyl primary amines having 18–24 carbon atoms in sufficient amount to solubilize said (N-stearoyl) sarcosine, in the mineral oil, the said alkyl amines being soluble in the mineral oil but insoluble in water.

15. A rust-preventive composition adapted to use in the presence of water, in systems containing metal susceptible of corrosion by said water, comprising a mineral oil and minor amounts of (N-lauroyl) sarcosine, as little as a fraction of 1% but in a sufficient quantity to inhibit the corrosion of said metal to a great degree when compared with the check, and alkyl primary amines having 18–24 carbon atoms in sufficient amount to solubilize said (N-lauroyl) sarcosine, in the mineral oil, the said alkyl amines being soluble in the mineral oil but insoluble in water.

16. A rust-preventive composition adapted to use in the presence of water, in systems containing metals susceptible of corrosion by said water, comprising a mineral oil and amounts of (N-stearoyl) alanine of the alpha and beta types of as little as a fraction of 1% but in sufficient quantity to inhibit the corrosion of said metal to

a great degree when compared with the check, and alkyl primary amines having 18–24 carbon atoms in sufficient amount to solubilize said (N-stearoyl) alanine, in the mineral oil, the said alkyl amines being soluble in the mineral oil but insoluble in water.

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