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CORROSION INHIBITORS

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This invention relates to corrosion inhibitors, and more particularly to an inhibitor for use in preventing corrosion of metal surfaces by water, associated in relatively small quantities, with organic materials.

In the manufacture, handling, transportation and/or use of various organic substances, corrosion problems occur due to the presence of varying amounts of water in solution or in suspension in the organic substances. Illustrative organic substances include hydrocarbon distillates as gasoline, jet fuel, kerosene, lubricating oil, fuel oil, diesel oil, crude oil, etc. Other specific oils include cutting oils, soluble oils, slushing oils, rolling oils, etc. which may be of mineral, animal or vegetable origin. Various coating compositions include grease, waxes, household oils, paints, lacquers, etc. Other organic substances include alcohols, ketones, esters, ethers, dioxane, amino compounds, amides, etc. In spite of all reasonable and practical precautions which are taken to avoid the presence of water, an appreciable quantity of water separation is found as a film or in minute droplets in the pipe line or on container walls or even in small pools at the bottom of the container. This results in corrosion of the metal surfaces and contamination of the organic substance by the corrosion products.

A recently proposed corrosion inhibitor of especial effectiveness is an alkyl acid phosphate salt of an N-alkyl-diaminoalkane, and particularly such a salt in which at least one of the alkyl groups constituting the ester portion of the acid phosphate contains at least 6 and preferably at least 8 carbon atoms. Particularly effective corrosion inhibitors include the mono-octyl acid orthophosphate salt of N-tallow-1,3-diaminopropane, dioctyl acid orthophosphate salt of N-tallow-1,3-diaminopropane, mixture of mono- and dioctyl acid orthophosphate salts of N-tallow-1,3-diaminopropane, similar salts or mixed salts in which the alkyl substituent of the acid orthophosphate comprises nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, octadecyl, nonadecyl, eicosyl, etc.

Other suitable corrosion inhibitors comprise alkyl acid orthophosphate salts of N-alkyl-1,3-diaminopropanes in which the alkyl group is derived from lauric acid, coconut fatty acid, soya fatty acid, etc. and thus may be designated as an alkyl acid orthophosphate salt of N-lauryl-1,3-diaminopropane, N-coco-1,3-diaminopropane, N-soya-1,3-diaminopropane, etc.

The N-tallow-1,3-diaminopropane contains mostly 16 to 18 carbon atoms per alkyl group, although it contains a small amount of 14 carbon atom alkyl group. The N-coco-1,3-diaminopropane contains alkyl groups having from 8 to 18 carbon atoms, predominating in 12 and 14 carbon atom alkyl groups. The soya-1,3-diaminopropane predominates in alkyl groups containing 18 carbon atoms per group, although it contains a small amount of a 16 carbon atom alkyl group.

While the N-alkyl-1,3-diaminopropanes are preferred, it is understood that other suitable N-alkyl-diaminoal-

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kanes may be employed including, for example, N-alkyl-1,2-diaminoethanes, N-alkyl-1,2-diaminopropanes, N-alkyl-1,2-diaminobutanes, N-alkyl-1,3-diaminobutanes, N-alkyl-1,4-diaminobutanes, N-alkyl-1,2-diaminopentanes, N-alkyl-1,3-diaminopentanes, N-alkyl-1,4-diaminopentanes, N-alkyl-1,5-diaminopentanes, N-alkyl-1,2-diaminohexanes, N-alkyl-1,3-diaminohexanes, N-alkyl-1,4-diaminohexanes, N-alkyl-1,5-diaminohexanes, N-alkyl-1,6-diaminohexanes, etc.

While the alkyl acid orthophosphate salts are preferred, in some cases the alkyl acid pyrophosphate salts may be employed. These alkyl acid pyrophosphate salts likewise contain at least 6 and preferably at least 8 carbon atoms in at least one of the alkyl groups and these are salts of the N-alkyldiaminoalkanes hereinbefore set forth.

It is understood that the various alkyl acid orthophosphate and alkyl acid pyrophosphate salts of N-alkyldiaminoalkanes are not necessarily equivalent in the same or different substrate. However, all of them will show some effectiveness as a corrosion inhibitor and may be used in accordance with the present invention.

As hereinbefore set forth, the alkyl acid orthophosphate and pyrophosphate salts of the N-alkyl-diaminoalkanes are effective corrosion inhibitors. However, in some cases, they do not meet the military specifications for water tolerance. The present invention is directed to a corrosion inhibitor composition, and use thereof, comprising the alkyl acid phosphate salts of N-alkyl-diaminoalkanes containing a minor proportion of a specific mixed condensation product which permits the inhibitor to pass the water tolerance test. As will be shown by the following examples, the condensation product is prepared by the use of particular reactants and also by the use of critical concentrations of these reactants. It is unexpected that the condensation product formed through the use of these critical concentrations will yield an inhibitor composition which passes the water tolerance test, in view of the fact that condensation products using concentrations outside the specified range are unsatisfactory.

In one embodiment the present invention relates to a corrosion inhibitor composition comprising an alkyl acid phosphate salt of an N-alkyl-diaminoalkane and the mixed condensation product of an N-alkyl-diethanolamine and N-methyl-diethanolamine with the reaction product of a terpene and a compound selected from the group consisting of an alpha, beta-unsaturated polycarboxylic acid, anhydride and ester thereof, said N-alkyldiethanolamine containing 8 to 18 carbon atoms in the alkyl group and being utilized in said condensation in a concentration of from about 0.2 to about 0.4 equivalent per 1 equivalent of said reaction product.

In another embodiment the present invention relates to a non-corrosive composition of matter comprising an organic material coming in contact with water during the useful life thereof, said organic material containing dissolved therein a corrosion inhibitor composition as herein set forth.

From the preceding description, it will be noted that the corrosion inhibitor comprises an alkyl acid phosphate salt of an N-alkyl-diaminoalkane together with a particular mixed condensation product. The mixed condensation product is formed by the mixed condensation of a particular N-alkyl-diethanolamine in a critical concentration and N-methyl-diethanolamine with the reaction product of a terpene and an alpha, beta-unsaturated polycarboxylic acid, anhydride or ester thereof.

The N-alkyl-diethanolamine contains from about 8 to about 18 and preferably 16 to 18 carbon atoms in the alkyl group. A particularly preferred N-alkyl-diethanolamine is N-tallow-diethanolamine which, as hereinbefore set forth, contains from 14 to 18 carbon atoms in the

alkyl group, mostly 16 to 18 carbon atoms. Other N-alkyl-diethanolamines include N-coco-diethanolamine and N-soya-diethanolamine which, as hereinbefore set forth, contain mixed alkyl groups. While these mixed alkyl groups generally are preferred because of their availability commercially at reasonable prices, it is understood that the N-alkyl-diethanolamine may be synthesized for use in accordance with the present invention. These N-alkyl-diethanolamines include N-octyl-diethanolamine, N-nonyl-diethanolamine, N-decyl-diethanolamine, N-undecyl-diethanolamine, N-dodecyl-diethanolamine, N-tridecyl-diethanolamine, N-tetradecyl-diethanolamine, N-pentadecyl-diethanolamine, N-hexadecyl-diethanolamine, N-heptadecyl-diethanolamine, N-octadecyl-diethanolamine, etc.

As hereinbefore set forth, the N-alkyl-diethanolamine is used in a critical concentration. It is also essential that the condensation is effected using N-methyl-diethanolamine along with the N-alkyl-diethanolamine mentioned above. One equivalent of the total of the two N-alkyl-diethanolamines is condensed with one equivalent of the reaction product of the terpene and acid, anhydride or ester. Therefore, by establishing the critical concentration of the N-alkyl-diethanolamine as between 0.2 and 0.4 equivalent, this establishes the concentration of the N-methyl-diethanolamine as between 0.6 and 0.8 equivalent per one equivalent of the terpene reaction product.

The reaction product of the terpene and alpha,beta-unsaturated polycarboxylic acid, anhydride or ester will comprise primarily the anhydride but the acid and/or ester also will be present. Any suitable terpenic compound may be reacted with any suitable alpha,beta-unsaturated polycarboxylic acid, anhydride or ester to form the reaction product for subsequent condensation with the N-alkyl-diethanolamines. In one embodiment a terpene hydrocarbon having the formula $C_{10}H_{16}$ is employed, including alpha-pinene, beta-pinene, dipentene, d-limonene, l-limonene and terpinoline. These terpene hydrocarbons have boiling points ranging from about 150° to about 185° C. In another embodiment the terpene may contain three double bonds in monomeric form, including terpene as allo-o-cymene, o-cymene, myrcene, etc. Other terpenic compounds include alpha-terpinene, p-cymene, etc.

As hereinbefore set forth, the terpene is reacted with an alpha,beta-unsaturated polycarboxylic acid, anhydride or ester thereof. Any unsaturated polycarboxylic acid having a point of unsaturation between the alpha and beta carbon atoms may be employed. Illustrative unsaturated dicarboxylic acids include maleic acid, fumaric acid, citraconic acid, mesaconic acid, aconitic acid, itaconic acid. While the dicarboxylic acids are preferred, it is understood that alpha,beta-unsaturated polycarboxylic acids containing three, four or more carboxylic acid groups may be employed. Furthermore, it is understood that a mixture of alpha,beta-unsaturated polycarboxylic acids and particularly of alpha,beta-unsaturated dicarboxylic acids may be used.

While the alpha,beta-unsaturated polycarboxylic acid may be employed, advantages appear to be obtained in some cases when using the anhydrides thereof. Illustrative anhydrides include maleic anhydride, citraconic anhydride, aconitic anhydride, itaconic anhydride, etc. It is understood that a mixture of anhydrides may be employed and also that the anhydride may contain substituents and particularly hydrocarbon groups attached thereto. Furthermore, it is understood that the various anhydrides are not necessarily equivalent. Also, it is understood that esters of the alpha,beta-unsaturated polycarboxylic acids may be employed, the ester group being selected from alkyl, alkaryl, aralkyl, aryl and cycloalkyl substituents replacing one or more of the hydrogen atoms of the carboxylic acid groups.

The reaction of terpene and alpha,beta-unsaturated acid, anhydride or ester generally is effected at a tem-

perature of from about 150° to about 300° C., and preferably of from about 160° to about 200° C. The time of heating will depend upon the particular reactants and may range from 2 hours to 24 hours or more. When desired, a suitable solvent may be utilized. Following the reaction, impurities or unreacted materials may be removed by vacuum distillation or otherwise, to leave a resinous product which may be a viscous liquid or a solid.

A terpene-maleic anhydride reaction product is available commercially under the trade name of "Petrex Acid." This acid is a stringy, yellow-amber colored mass and is mostly dibasic. It has an acid number of approximately 530, a molecular weight of approximately 215 and a softening point of 40°-50° C.

As hereinbefore set forth, one equivalent of the total of the two N-alkyl-diethanolamines is reacted with one equivalent of the terpene reaction product. The equivalent of the N-alkyl-diethanolamines is determined by the sum of the hydroxyl groups in the two N-alkyl-diethanolamines. The equivalent of the terpene reaction product is determined by the number of carboxylic acid or potential carboxylic acid groups.

The condensation of the N-alkyl-diethanolamines and terpene reaction product is effected in any suitable manner. The condensation generally is effected at a temperature above about 80° C. and preferably at a higher temperature which usually will not exceed about 200° C., although higher or lower temperatures may be employed under certain conditions. The exact temperature will depend upon whether a solvent is used and, when employed, on the particular solvent. For example, with benzene as the solvent, the temperature will be in the order of 80° C., with toluene the temperature will be in the order of 120° C., and with xylene in the order of 150°-155° C. Other preferred solvents include cumene, naphtha, etc. Any suitable amount of the solvent may be employed but preferably should not comprise a large excess because this will tend to lower the reaction temperature and slow the reaction. Water formed during the reaction may be removed in any suitable manner including, for example, by operating under reduced pressure, by removing an azeotrope of water-solvent, by distilling the condensation product at an elevated temperature, etc. A higher temperature may be utilized in effecting the reaction in order to remove the water as it is being formed.

In another embodiment of the invention the condensation product may contain N,N-diethyl-ethanolamine. The N,N-diethyl-ethanolamine may replace up to about 1/3 of the N-methyl-diethanolamine. Thus, the total of N-methyl-diethanolamine and N,N-diethyl-ethanolamine will be employed in a concentration within the range of 0.6 to 0.8 equivalent per 1 equivalent of the terpene reaction product.

The mixed condensation product of the two N-alkyl-diethanolamines or of the two N-alkyl-diethanolamines and N,N-diethyl-ethanolamine is incorporated in the alkyl acid phosphate salt of N-alkyl-diaminoalkane in a concentration of from about 0.1 to about 5% by weight of the salt, although higher concentrations up to 25% may be used in some cases. For ease of handling, the inhibitor composition is prepared as a solution in a suitable solvent. In one method, the alkyl phosphate salt of N-alkyl-diaminoalkane is prepared as a solution in a solvent, and the mixed condensation product of the N-alkyl-diethanolamines or N-alkyl-diethanolamines and N,N-diethyl-ethanolamine with the terpene reaction product is incorporated in the solution. Any suitable solvent may be employed and preferably comprises a hydrocarbon distillate or hydrocarbon fraction including benzene, toluene, xylene, cumene, etc. The solution may contain from about 10 to 80% by weight of active constituents and preferably from about 35 to about 70% by weight of active constituents.

The amount of inhibitor composition to be incorporated will depend upon the particular organic substrate in which

it is to be used. In general, the inhibitor composition will be used in a concentration of less than about 5% by weight and thus may range from about 0.0001% to about 5% by weight and still more preferably within the range of from about 0.001% to about 1% by weight of the organic substance. It is understood that this inhibitor composition may be used along with other additives used for specific purposes in the organic substrate and, when desired, the inhibitor composition of the present invention may be admixed with the other additive or additives and marketed as a single commodity of multiple purposes.

As hereinbefore set forth, the inhibitor composition of the present invention may be utilized in any organic substance containing or contacting water and causing corrosion of metals. In one embodiment the inhibitor composition is added directly to the organic substance and intimately mixed to obtain distribution of the inhibitor composition in the organic substrate. When used in plant equipment, the inhibitor composition of the present invention may be introduced into the fractionator or pipe line of the plant equipment, to thereby prevent corrosion of the plant equipment. Generally, a sufficient concentration of the inhibitor composition will be employed so that a portion of the inhibitor composition is retained in the organic substance itself and serves to retard corrosion of metal piping or containers through or into which the organic substance is subsequently passed.

The following examples are introduced to illustrate further the novelty and utility of the present invention but not with the intention of unduly limiting the same.

EXAMPLE I

This example illustrates the effectiveness of a mixture of mono- and dioctyl acid orthophosphate salt of N-tallow-1,3-diaminopropane as a corrosion inhibitor. The salt was prepared as a 50% by weight solution in toluene.

This salt was tested by the ASTM steam turbine oil corrosion test (ASTM D665-52T), which test has been found to correlate with the results obtained in the storage of oil in storage tanks. In this test, 300 cc. of Nujol, to which 30 cc. of synthetic sea water is added, is placed in a beaker open to the atmosphere. A highly polished mild carbon steel rod is suspended in the oil-water suspension, heated to and maintained at 140° F., with stirring, for 48 hours. The steel rod then is inspected and the corrosion reported as light, medium or heavy. In addition, the portion of the rod covered with rust is also reported as a number of $\frac{1}{10}$ of the rod covered with rust, as well as also reporting the extent of pitting.

The results of this test is shown in the following table, along with the results obtained when not using any inhibitor. The percent inhibitor used is on the basis of active constituents, exclusive of solvent.

Table I

Inhibitor	Corrosion, 1/10	Coverage	Pitting
None.....	Heavy.....	10	Deep.
0.01%.....	Clean.....	0	None.

EXAMPLE II

As hereinbefore set forth, the corrosion inhibitor of Example I is very effective but does not pass the water tolerance test as specified in MIL-I-25017. According to this test, the maximum allowable concentration of corrosion inhibitor (20 lb. per 1000 barrels of oil) is incorporated in iso-octane. More specifically, a mixture of 80 ml. of iso-octane and 20 ml. of water is prepared and the inhibitor is incorporated therein. The procedure then is to shake the mixture for 2 minutes, allow it to stand for 5 minutes, after which it is inspected. In order to pass this test, there must be a clear break. In other words, there must be no cloudiness at the interface of

oil and water. The results are reported as either "pass" or "fail."

When tested in the above manner, the corrosion inhibitor of Example I will not pass this test. On the other hand, a sample of the same inhibitor solution containing 1.5% by weight of the mixed condensation product hereinafter described passed the water tolerance test.

The mixed condensation product referred to above was prepared by the condensation of 0.25 equivalent of N-tallow-diethanolamine and 0.75 equivalent of N-methyldiethanolamine with one equivalent of Petrex Acid. This condensation was effected using xylene as the solvent, after which the xylene was removed by distilling under vacuum.

EXAMPLE III

A similar water tolerance test for jet fuel is conducted in substantially the same manner except that JP-4 jet fuel is used. This test is designated as MIL-F-5624.

When a sample of the inhibitor described in Example I was tested in this manner, it failed to pass the water tolerance test for JP-4 fuel. On the other hand, a sample containing 1.5% by weight of the inhibitor solution of the mixed condensation product described in Example II did pass the JP-4 water tolerance test.

EXAMPLE IV

A sample of the inhibitor described in Example I and a sample of the inhibitor described in Example II were each separately tested according to the method of specification MIL-I-25017. This test is substantially the same as the ASTM Steam Turbine Oil Corrosion Test hereinbefore described.

When evaluated according to this test, the results when using the phosphate inhibitor of Example I and the inhibitor composition of Example II were substantially the same. This demonstrates that the incorporation of the mixed condensation product did not deleteriously affect the potency of the corrosion inhibitor.

EXAMPLE V

As previously set forth, the N-alkyl-diethanolamine containing 8 to 18 carbon atoms in the alkyl group and the N-methyl-diethanolamines must be used in critical concentrations in the condensation with Petrex Acid. This is illustrated by the following data, in which concentrations of these N-alkyl-diethanolamines within and outside of the ranges herein specified were employed. In most cases it is required that the corrosion inhibitor be compatible with other additives used in the organic substrate, and the present tests were made using an equivalent amount of the inhibitor composition of the present invention and a commercial additive approved by the military specifications, each additive being used in a concentration equivalent to 10 lbs. per 1000 barrels of oil or a total of 20 lbs. per 1000 barrels of oil. The results of these tests are shown in the following table.

Table II

Condensation of:

Petrex Acid, Equival- ents	N-tallow- di-ethanol- amine, Equival- ents	N-methyl- di-ethanol- amine, Equival- ents	Water Tol- erance Test, Iso- octane
1	1	-----	fail.
1	0.5	0.5	fail.
1	0.2	0.8	pass.
1	0.25	0.75	pass.

From the data in the above table, it will be noted that the mixtures containing 0.5 equivalent or more of N-tallow-diethanolamine failed to pass the water tolerance test of this example, whereas the mixtures containing

0.2 and 0.25 equivalent thereof did pass the water tolerance test.

EXAMPLE VI

As hereinbefore set forth, a portion of the N-methyl-diethanolamine may be replaced by N,N-diethyl-ethanolamine. This is illustrated in the present example, in which the condensation was effected using 1 equivalent of Petrex Acid, 0.2 equivalent of N-tallow-diethanolamine, 0.7 equivalent of N-methyl-diethanolamine and 0.1 equivalent of N,N-diethyl-ethanolamine. When tested in accordance with the procedure described in Examples II and III, using 1.5% by weight of the mixed condensation product in the corrosion inhibitor described in Example I, the oil containing the inhibitor composition passed both water tolerance tests.

I claim as my invention:

1. A corrosion inhibitor composition comprising (1) an alkyl acid phosphate salt of an N-alkyl-diaminoalkane in which the alkyl group of the acid phosphate contains from about 6 to about 20 carbon atoms and the alkyl group of said diaminoalkane contains from about 8 to about 18 carbon atoms, and (2) from about 0.1% to about 25% by weight, based on said salt, of the mixed condensation product of (a) one equivalent of an N-alkyl-diethanolamine containing from 8 to 18 carbon atoms in the alkyl group and N-methyl-diethanolamine with (b) one equivalent of the reaction product of a terpene having the formula $C_{10}H_{16}$ and a boiling point ranging from about 150° to about 185° C. with a compound selected from the group consisting of an alpha, beta-unsaturated polycarboxylic acid, anhydride and ester thereof, said terpene and said compound having been reacted at a temperature of from about 150° to about 300° C. to form said reaction product, said N-alkyl-diethanolamine and N-methyl-diethanolamine having been condensed with said reaction product at a temperature of from about 80° to about 200° C. to form said mixed condensation product, and said N-alkyl-diethanolamine being utilized in the condensation in a concentration of from about 0.2 to about 0.4 equivalents per 1 equivalent of said reaction product.

2. A corrosion inhibitor composition comprising (1) an alkyl acid phosphate salt of an N-alkyl-diaminoalkane in which the alkyl group of the acid phosphate contains from about 6 to about 20 carbon atoms and the alkyl group of said diaminoalkane contains from about 8 to about 18 carbon atoms, and (2) from about 0.1% to about 25% by weight, based on said salt, of the mixed condensation product of (a) one equivalent of an N-alkyl-diethanolamine containing from 8 to 18 carbon atoms in the alkyl group and N-methyl-diethanolamine with (b) one equivalent of the reaction product of maleic anhydride with a terpene having the formula $C_{10}H_{16}$ and a boiling point ranging from about 150° to about 185° C., said terpene and maleic anhydride having been reacted at a temperature of from about 150° to about 300° C. to form said reaction product, said N-alkyl-diethanolamine and N-methyl-diethanolamine having been condensed with said reaction product at a temperature of from about 80° to about 200° C. to form said mixed condensation product, and said N-alkyl-diethanolamine being utilized in the condensation in a concentration of from about 0.2 to about 0.4 equivalents per 1 equivalent of said reaction product.

3. A corrosion inhibitor composition comprising (1) an alkyl acid phosphate salt of an N-alkyl-diaminoalkane in which the alkyl group of the acid phosphate contains

from about 6 to about 20 carbon atoms and the alkyl group of said diaminoalkane contains from about 8 to about 18 carbon atoms, and (2) from about 0.1% to about 25% by weight, based on said salt, of the mixed condensation product of (a) one equivalent of N-tallow-diethanolamine and N-methyl-diethanolamine with (b) one equivalent of the reaction product of maleic anhydride with a terpene having the formula $C_{10}H_{16}$ and a boiling point ranging from about 150° to about 185° C., said terpene and maleic anhydride having been reacted at a temperature of from about 150° to about 300° C. to form said reaction product, said N-tallow-diethanolamine and N-methyl-diethanolamine having been condensed with said reaction product at a temperature of from about 80° to about 200° C. to form said mixed condensation product, and said N-tallow-diethanolamine being utilized in the condensation in a concentration of from about 0.2 to about 0.4 equivalent per 1 equivalent of said reaction product.

4. A corrosion inhibitor composition comprising (1) an octyl acid phosphate salt of N-tallow-1,3-diaminopropane and (2) from about 0.1% to about 25% by weight, based on said salt, of the mixed condensation product of (a) one equivalent of N-tallow-diethanolamine and N-methyl-diethanolamine with (b) one equivalent of the reaction product of maleic anhydride with a terpene having the formula $C_{10}H_{16}$ and a boiling point ranging from about 150° to about 185° C., said terpene and maleic anhydride having been reacted at a temperature of from about 150° to about 300° C. to form said reaction product, said N-tallow-diethanolamine and N-methyl-diethanolamine having been condensed with said reaction product at a temperature of from about 80° to about 200° C. to form said mixed condensation product, and said N-tallow-diethanolamine being utilized in the condensation in a concentration of from about 0.2 to about 0.4 equivalent per 1 equivalent of said reaction product.

5. A corrosion inhibitor composition comprising (1) a mixture of mono- and dioctyl acid phosphate salts of N-tallow-1,3-diaminopropane and (2) from about 0.1% to about 25% by weight, based on said salts, of the mixed condensation product of (a) one equivalent of N-tallow-diethanolamine and N-methyl-diethanolamine with (b) one equivalent of the reaction product of maleic anhydride with a terpene having the formula $C_{10}H_{16}$ and a boiling point ranging from about 150° to about 185° C., said terpene and maleic anhydride having been reacted at a temperature of from about 150° to about 300° C. to form said reaction product, said N-tallow-diethanolamine and N-methyl-diethanolamine having been condensed with said reaction product at a temperature of from about 80° to about 200° C. to form said mixed condensation product, and said N-tallow-diethanolamine being utilized in the condensation in a concentration of from about 0.2 to about 0.4 equivalent per 1 equivalent of said reaction product.

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