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ANTI-CORROSION COMPOSITIONS AND MINERAL OIL COMPOSITIONS CONTAINING THE SAME

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13 Claims. (Cl. 252—49.9)

This invention relates to new anti-corrosion compositions and to mineral oil compositions containing the same. More particularly, the invention relates to a combination of oil-soluble amines and the substantially neutral addition products of olefin oxides and dialkyl orthophosphoric acids, and to mineral oil compositions containing such mixtures and having improved corrosion inhibiting properties.

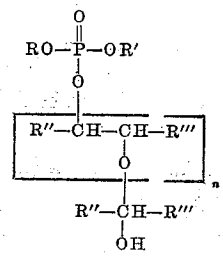
As is well known in the art, uncompounded mineral oils are often deficient in one or more respects. For example, most mineral oils that do not contain improvement agents fail to provide adequate protection against rust and corrosion. Fuels and lubricants for internal combustion engines and for gas or steam turbines are examples of materials which may desirably have incorporated therein improvement agents which impart corrosion inhibiting properties thereto. A high resistance to rust or corrosion is important in the case of fuels or lubricants which contact metal surfaces in the presence of water. Failure to provide sufficient resistance to rust or corrosion by a particular mineral oil may result in extensive wear or damage to costly, finely machined, moving parts and/or damage to storage and transport facilities, etc.

We have found that the corrosion inhibiting properties of mineral oils are improved by incorporation therein of minor amounts of a combination of (*a*) an oil-soluble aliphatic amine containing 8 to 18 carbon atoms and (*b*) a substantially neutral addition product of a C_2 to C_4 olefin oxide and a dialkyl orthophosphoric acid whose alkyl groups contain 5 to 8 carbon atoms, where the weight ratio of said amine and said addition product is about 1:50 to about 1:1. The present invention relates to mineral oil compositions containing such corrosion inhibiting additive combinations and to the additive combinations as such. The combinations of this invention include simple mixtures of amine and addition product, such as occur when the addition product has been fully reacted with alkylene oxide and exhibits a pH close to 6.8, reaction products such as occur when a small proportion of amine (e.g. 1 part) is used in conjunction with a substantially larger proportion (e.g. 10 parts, 20 parts) of an addition product which has a pH near 5 and still possesses some acidity, and mixtures of such simple mixtures and reaction products such as can occur when the amount of amine added is more than enough to neutralize any residual acidity exhibited by the addition product.

Any oil-soluble amine containing 8 to 18 carbon atoms can be used in the compositions of this invention. An example of a preferred oil-soluble amine is cocoamine. Cocoamine is a commercial mixture of straight chain primary aliphatic monoamines derived from coconut oil fatty acids and consisting mostly of n-dodecyl amine together with lesser proportions of n-octyl, n-decyl, n-tetradecyl, n-hexadecyl, n-octadecyl and n-octadecenyl amines. Individual amines of the class described, such as n-octyl and n-dodecyl amines, can also be used. Branched chain amines such as 1,1,3,3,-tetramethylbutyl amine and 1,1,3,3,5,5,-hexamethylhexyl amine are also suitable for the purposes of this invention. Amines other than primary amines can also be used but in such cases the amines should contain at least one hydrocarbon substituent capable of imparting oil-solubility to the amine. Examples of such amines are dicyclohexyl amine and N,N-dimethyl n-octyl amine, and triamyl amine.

The substantially neutral addition products of the class described herein are prepared as described in our copending application Serial No. 569,684. As described in that application the substantially neutral addition products are prepared by reacting an olefin oxide that contains 2 to 4 carbon atoms per molecule with a dialkyl ester of orthophosphoric acid whose alkyl groups contain 5 to 8 carbon atoms in proportions such as to provide a substantially neutral addition product, that is, one having a pH of about 5 to 6.8. Usually several mols of olefin oxide per mol of dialkyl acid orthophosphate will be required to produce a substantially neutral addition product. The actual number of mols required can vary somewhat with the identity of the olefin oxide and the dialkyl acid o-phosphate. The substantially neutral products can be conveniently obtained by addition of olefin oxide in small increments to the dialkyl acid o-phosphate under ordinary atmospheric conditions of temperature and pressure and determining the pH of the reaction mixture following the addition of each such increment. The reaction is regarded as complete when the reaction mixture possesses a pH of about 5 to 6.8. The neutralization reaction described is normally exothermic and takes place spontaneously at room temperature to the degree indicated.

The products resulting from the substantial neutralization of a dialkyl acid orthophosphate with an olefin oxide are usually light-colored, slightly viscous, oil-soluble liquids. These addition products will comprise principally alkyloloxyalkylene dialkyl orthophosphates having the generic formula:



where R and R′ are alkyl groups containing 5 to 8 carbon atoms, where R″ and R‴ are methyl, ethyl or hydrogen groups, the total number of carbon atoms in the R″ and R‴ groups being not greater than 2, and *n* is a number of 0 to 7, preferably 0 to 2 or 3. The class of compounds indicated above is considered representative of the principal components of the substantially neutral addition products described herein, but it will be appreciated that the reaction mixture undoubtedly contains a mixture of products. For example, the reaction mixture can contain reaction products of the formula indicated above where *n* is 0 to 7 or more. For this reason we prefer to refer to the phosphate addition agents described herein as substantially neutral "addition products" rather than as specific chemical compounds.

Specific examples of preferred addition products, the use of which is included by this invention, are the substantially neutral addition products of propylene oxide and di-(3-methylbutyl) acid o-phosphate, 3-methylbutyl, 2-ethylhexyl acid o-phosphate and di-(Oxo-octyl) acid o-phosphate. Specific components of these addition products are 2 - hydroxy-4,7-dioxa - 5,8 - dimethylnonyl di-(3-methylbutyl) o-phosphate, 2-hydroxy-4,7,10-trioxa-5,8,11-trimethyldodecyl,3-methylbutyl,2-ethylhexyl o-phosphate, and 2-hydroxy-4,7-dioxa-5,8-dimethylonyl di-(Oxo-octyl) o-phosphate. Examples of other substantially neutral addition products, the use of which is included by this invention, are the substantially neutral addition products of ethylene oxide, propylene oxide, and for example, 1,2-butylene oxide, and di-(2-ethylhexyl) acid o-phosphate, di-n-heptyl acid o-phosphate, di-t-octyl acid o-phosphate, and di-(dimethylhexyl) acid o-phosphate.

By "Oxo-octyl" is meant the octyl radical derived from Oxo-octyl alcohols. As is known, Oxo alcohols normally comprise mixtures of isomeric, mostly branched chain, primary monohydric alcohols produced by the Oxo synthesis process. The Oxo synthesis process as is well-known involves the catalytic hydroformylation of monoolefins, such as, for example, hexene and heptene, usually in the form of a mixture of isomers such as can be obtained by non-selective polymerization of the C_3, C_4 or mixed C_3 and C_4 monoolefins present in refinery gases, with a mixture of carbon monoxide and hydrogen, to obtain a mixture of oxygenated products, principally aldehydes, having one more carbon atom per molecule than the original monoolefin. The mixture of oxygenated products is then catalytically hydrogenated to produce Oxo alcohols.

The oil-soluble amines described above and the substantially neutral addition products described herein can be admixed in any convenient manner, each of these materials being completely miscible with the other in all proportions. As previously indicated, combinations possessing valuable corrosion inhibiting properties are obtained by the use of the above-described amine and the substantially neutral addition products in a range of proportions varying from about 1:50 to about 1:1 by weight. For example, excellent results have been obtained with combinations wherein the amine:alkylolpolyoxyalkylene dialkyl o-phosphate ratio was 1:20, 1:10, and 1:1.5, and this range of proportions is preferred. The corrosion inhibiting combinations of this invention normally exhibit a pH of about 5.5 to about 10. Combinations having a pH of about 7.0 to about 7.5 exhibit especially effective rust and corrosion inhibiting characteristics.

The preparation of a preferred class of corrosion inhibiting compositions according to this invention is illustrated by the following specific embodiments.

Example I

Phosphoric anhydride (P_2O_5) in the amount of 16.9 parts by weight was gradually added to 42.1 parts by weight of isoamyl alcohol, these quantities being equivalent to an approximate mol ratio of about one mol of anhydride per 4 mols of alcohol. The reaction proceeded spontaneously and exothermically in the early stages, the rate of addition of phosphoric anhydride being controlled to maintain a maximum reaction temperature of 120° F. When all of the phosphoric anhydride had been added the temperature of the reaction mixture was raised to a maximum of about 260° F. until all of the phosphoric anhydride went into solution. To the phosphate ester product of the initial reaction there was then added 36.2 parts by weight of propylene oxide. The mol ratio of reactants was about 3 mols of propylene oxide per mol of phosphate ester. This reaction was carried out under reflux with the propylene oxide being added in small increments down the tube of a water cooled reflux condenser. The rate of addition of propylene oxide was controlled so as to avoid formation of liquid slugs in the condenser tube. The addition of propylene oxide was commenced under atmospheric conditions of temperature and pressure. The reaction proceeded spontaneously with evolution of heat. The product of the foregoing reactions was a substantially neutral addition product of propylene oxide and di-(3-methylbutyl) acid o-phosphate, consisting chiefly of 2-hydroxy-4,7-dioxa - 5,8 - dimethylnonyl di-(3-methylbutyl) o-phosphate. To this product there was then added 4.8 parts by weight of cocoamine. This corresponded to a cocoamine:phosphate ester weight ratio of about 1:20. The composition prepared as described above had the following inspections:

Gravity, °API	8.1
Viscosity, SUV:sec., 100° F	115.5
Flash, OC °F	150
Pour: °F	—35
Appearance	Bright
Physical state:room temp	Liquid
Color, Saybolt	—16
Phosphorus, percent	7.34
Water by distillation:percent by weight, ASTM D 95–46 (Mod.)	1.0
Neutralization value, ASTM D 974–54T, total acid number	1.51
pH value, glass Calomel electrodes	9.5
Ash, percent	0.296

Example II

Another corrosion inhibiting composition according to this invention was prepared as indicated above using 49.3 parts by weight of Oxo-octyl alcohols prepared from mixed heptene isomers and consisting of mixed highly branched chain octyl alcohols, mostly dimethyl hexanols, 13.5 parts by weight of phosphorus pentoxide and 32.4 parts by weight of propylene oxide. These proportions corresponded to an approximate mol ratio of 4 mols of alcohol, 1 mol of phosphorus pentoxide and 6 mols of propylene oxide. The chief component of the product of these reactions was 2-hydroxy-4,7-dioxa-5,8-dimethylnonyl di-(Oxo-octyl) o-phosphate. To this product there was then added 4.8 parts by weight of cocoamine. The cocoamine:phosphate ester ratio in the resulting mixture was about 1:20. The composition prepared according to this example had the following inspections:

Gravity, ° API	12.3
Viscosity, SUV:sec., 100° F.	141.5
Flash, OC, ° F.	230
Pour: ° F.	—40
Appearance	Bright
Physical state, room temp.	Liquid
Color, ASTM union	1.0
Phosphorus, percent	7.22
Water by distillation, percent by wt., ASTM D 95–46 (Mod.)	1.50
Neutralization value, ASTM D 974–54T, total acid number	1.10
pH value, glass Calomel electrodes	8.1
Ash, percent	0.326

Example III

Another corrosion inhibiting composition according to this invention was prepared by reacting 28.5 parts by weight of propylene oxide with 65.0 parts by weight of a previously prepared 3-methylbutyl-2-ethylhexyl acid o-phosphate. These quantities corresponded to a mol ratio of about 4 mols of propylene oxide per mol of phosphate ester. The principal component to the product of the foregoing reaction was 2-hydroxy-4,7,10-trioxa-5,8,11 - trimethydodecyl,3 - methylbutyl,2 - ethylhexyl o-phosphate. To the product of the foregoing reaction there was added 6.5 parts by weight of cocoamine. The cocoamine:addition product weight ratio in this composition corresponded to about 1:10. The corrosion in-

hibiting mixture prepared according to this example had the following inspections:

Gravity, °API	15.8
Viscosity, SUV: sec., 100° F.	107.2
Flash, OC, ° F.	185
Pour, ° F.	−25
Appearance	Bright
Physical state, room temp.	Liquid
Color, ASTM union	2.75
Phosphorus, percent	4.97
Water by distillation, percent by wt., ASTM D 95–46 (Mod.)	0.1
Neutralization value, ASTM D 974–54T, Total acid number	4.73
pH value, glass Calonmel electrodes	8.0
Ash, percent	1.03

The corrosion inhibiting compositions described in the foregoing specific embodiments are illustrative only, and the invention is not limited thereto. Satisfactory results can be obtained by the substitution in the foregoing examples of other amines disclosed herein and other alkylolpolyoxyalkylene dialkyl o-phosphates disclosed herein in the same or equivalent proportions.

The amine:alkylolpolyoxyalkylene dialkyl o-phosphate combinations of this invention are useful when incorporated in mineral oils in minor proportions, sufficient to confer corrosion inhibiting properties upon the composition. For example, satisfactory results will be obtained by the use of about 0.01 to about 0.5 percent of the combination based on the weight of the mineral oil composition. Especially effective results will ordinarily be obtained by the use of about 0.03 to about 0.2 percent by weight of the corrosion inhibiting combinations disclosed herein, and this constitutes the preferred range of proportions.

The corrosion inhibiting compositions of this invention can be used in a wide variety of mineral oil compositions. For example, excellent results have been obtained by the use of the herein described corrosion inhibiting combinations in gasoline and lubricating oils. These corrosion inhibiting combinations can also be employed in conjunction with furnace oil, diesel fuel, slushing oils and greases. The herein described corrosion inhibiting combinations can also be incorporated in mineral oil products, such as light naphtha, which are employed neither as a fuel nor as a lubricant. In such instances the compositions find use in the coating art. In the use of the combinations of this invention in coating compositions, it may be desirable to employ the mixtures in substantially greater concentrations than are normally utilized in fuels and lubricants.

Several gasoline compositions having improved corrosion inhibiting characteristics were made up by incorporation of various amounts of the compositions of

Examples I, II and III in a base gasoline having the following typical inspections:

Gravity, ° API	68.3
Specific gravity	0.708
Sulfur (lamp), percent	0.03
Existent gum, mg./100 ml.	1
Potential gum, mg./100 ml.	3
Knock rating, octane No.:	
Motor	92
Aviation lean mixture	91.2
Aviation rich mixture	98.7
TEL, ml./gal.	4.00
Reid vapor pressure, lbs.	6.7
Distillation, ° F.:	
Over point	126
End point	285
10 percent evap. at	145
50 percent	180
90 percent	224

The tetraethyl lead in the foregoing gasoline was added in the form of a commercial aviation mix containing 1.9 cc. of ethylene dibromide and a dye. In addition, the foregoing base gasoline contained about 8.4 pounds per thousand barrels of 2,6-di-t-butyl-4-methylphenol, an oxidation inhibitor.

The corrosion inhibiting properties of the foregoing compositions were demonstrated by subjecting these compositions to an accelerated rusting test. Briefly, this test involved immersion of a polished cylindrical steel specimen in a stirred mixture of the gasoline under test with 10 percent by volume of distilled water, at a temperature of 80° F. for 4 hours. At the conclusion of the test the steel specimen was examined for rust and rated according to the following scale: no rust=0, 1 to 5 percent rust=1, 6 to 25 percent rust=2, 26 to 50 percent rust=3, and more than 50 percent rust=4.

The make-up of the gasoline compositions tested and the results of these tests are summarized in the following table:

	Uninhibited Base Gasoline	Composition IV	Composition V	Composition VI
Make-up:				
Base Gasoline, Percent by Volume	100	100	100	100
Inhibitor Added—				
Example I Inhibitor Composition:				
Lbs./1,000 Bbls.		87.9		
Weight Percent		0.036		
Example II Inhibitor Composition:				
Lbs./1,000 Bbls.			105	
Weight Percent			0.042	
Example III Inhibitor Composition:				
Lbs./1,000 Bbls.				126
Weight Percent				0.05
Inspections:				
Existent gum:mg./100 Ml., ASTM D 381–50	0	1	1	1
Oxidation Stability:Min., ASTM D 525–49	1,440+	1,440+	1,440+	1,440+
Rust Preventive Test, Distilled Water, 4 Hr., 80° F.—Rating	4	0	0	0

The results set forth in the foregoing table clearly demonstrate the marked improvement in corrosion inhibiting properties obtained by the use of small amounts of the inhibitor compositions of this invention. The results obtained in these examples are even more remarkable when it is considered that the use in the same base gasoline of the identical amounts of the same alkylolpolyoxyalkylene dialkyl o-phosphates, without the addition of any amine, fails to produce any improvement in corrosion inhibiting properties over those of the uninhibited base gasoline. Also, the cocoamine, as such, when tested in a sample of the same kind of gasoline in amounts of 5 and 100 pounds per thousand barrels, produced, respectively, 4 hour rust ratings of 2 and 1 and 24 hour rust ratings, respectively, of 4 and 3, as

compared with 4 and 24 hour rust ratings of 2 and 4, respectively, for the uninhibited gasoline. Obviously, the small amount of amine present in compositions IV, V and VI could not alone account for the remarkable improvement obtained.

An additional corrosion inhibiting mineral oil composition according to this invention was prepared by incorporating in a lubricating oil a small amount of a corrosion inhibiting mixture containing a 1:4 mixture of cocoamine and a substantially neutral addition product of propylene oxide and 3-methylbutyl,2-ethylhexyl acid o-phosphate. The uninhibited lubricating oil stock used in this composition was a highly refined paraffinic lubricating oil having the following inspections:

Gravity: ° API	31.7
Viscosity, SUV:Sec.:	
70° F.	336
210° F.	43.8
Flash, OC: ° F.	425
Viscosity index	110

The foregoing composition was subjected to the conditions of an accelerated rust test carried out similarly as that described above, except that the test composition was maintained at a temperature of about 140° F. and except that the test was continued for 24 hours. Duplicate tests were carried out using distilled water and synthetic sea water (ASTM D 665–52T). At the conclusion of each test the steel test specimen was examined for rust and rated as passing if no rust appeared and failing if any rust was found.

The make-up of the foregoing composition and the test results are summarized in the following table:

	Uninhibited Oil	Composition VII
Make-up:		
Uninhibited Lubricating Oil, Percent by Volume	100	100
Inhibitor Added: Percent by Weight—		
1:4 Wt. Ratio Mixture of Cocoamine and Subst. Neut. Add. Product of Propylene Oxide and 3-Methylbutyl,2-ethylhexyl Acid o-phosphate (85% Mineral Oil Concentrate)		0.15
Inspections:		
Rust Preventive Test, 140° F., 24 Hrs.—		
Distilled Water	Fail	Pass
Synthetic Sea Water	Fail	Pass

The foregoing data clearly demonstate the beneficial results obtainable with the corrosion inhibiting compositions of this invention.

Obviously many modifications or variations of the invention as hereinabove set forth can be resorted to without departing from the spirit and scope thereof. Accordingly, only such limitations should be imposed as are contained in the claims appended hereto.

We claim:

1. A corrosion inhibiting composition consisting essentially of a combination of an oil-soluble aliphatic amine containing 8 to 18 carbon atoms and a substantially neutral addition product of an olefin oxide that contains 2 to 4 carbon atoms per molcule and a dialkyl acid o-phosphate whose alkyl groups contain 5 to 8 carbon atoms, the weight ratio of said amine and said substantially neutral addition product being about 1:50 to about 1:1.

2. The composition of claim 1 wherein the pH of the composition is about 7.0 to about 7.5.

3. A corrosion inhibiting composition consisting essentially of a combination of (*a*) mixed oil-soluble n-alkyl amines containing 8 to 18 carbon atoms and (*b*) a substantially neutral addition product of propylene oxide and di-(3-methylbutyl) acid o-phosphate, where the weight ratio of said amines and said substantially neutral addition product is about 1:50 to about 1:1.

4. A corrosion inhibiting composition consisting essentially of a combination of (*a*) mixed oil-soluble n-alkyl amines containing 8 to 18 carbon atoms and (*b*) a substantially neutral addition product of propylene oxide and di-(Oxo-octyl) acid o-phosphate, where the weight ratio of said amines and said substantially neutral addition product is about 1:50 to about 1:1.

5. A corrosion inhibiting composition consisting essentially of a combination of (*a*) mixed oil-soluble n-alkyl amines containing 8 to 18 carbon atoms and (*b*) a substantially neutral addition product of propylene oxide and 3-methylbutyl,2-ethylhexyl acid o-phosphate, where the weight ratio of said amines and said substantially neutral addition product is about 1:50 to about 1:1.

6. A corrosion inhibiting composition adapted to be incorporated in minor proportions in mineral oils, consisting essentially of a combination of an oil-soluble aliphatic amine containing 8 to 18 carbon atoms and a substantially neutral addition product of an olefin oxide that contains 2 to 4 carbon atoms per molecule and a dialkyl acid o-phosphate whose alkyl groups contain 5 to 8 carbon atoms, the weight ratio of said amine and said substantially neutral addition product being about 1:50 to about 1:1.

7. A mineral oil composition selected from the group consisting of fuel and lubricant compositions, comprising a major amount of a mineral oil and a minor amount, sufficient to confer corrosion inhibiting properties upon the composition, of a combination of an oil-soluble aliphatic amine containing 8 to 18 carbon atoms and a substantially neutral addition product of an olefin oxide that contains 2 to 4 carbon atoms per molecule and a dialkyl acid o-phosphate whose alkyl groups contain 5 to 8 carbon atoms, the weight ratio of said amine and said substantially neutral addition product being about 1:50 to about 1:1.

8. The composition of claim 7 wherein said minor amount is about 0.01 to about 0.5 percent by weight of the composition.

9. The composition of claim 7 wherein said mineral oil is a lubricating oil.

10. The composition of claim 7 wherein said mineral oil is gasoline.

11. A mineral oil composition selected from the group consisting of fuel and lubricant compositions, comprising a major amount of a mineral oil and a minor amount, sufficient to confer corrosion inhibiting properties upon the composition, of a combination of (*a*) mixed oil-soluble n-alkyl amines containing 8 to 18 carbon atoms and (*b*) a substantially neutral addition product of propylene oxide and di-(3-methylbutyl) acid o-phosphate, where the weight ratio of said amines and said substantially neutral addition product is about 1:50 to about 1:1.

12. A mineral oil composition selected from the group consisting of fuel and lubricant compositions, comprising a major amount of a mineral oil and a minor amount, sufficient to confer corrosion inhibiting properties upon the composition, of a combination of (*a*) mixed oil-soluble n-alkyl amines containing 8 to 18 carbon atoms and (*b*) a substantially neutral addition product of propylene oxide and di-(Oxo-octyl) acid o-phosphate, where the weight ratio of said amines and said substantially neutral addition product is about 1:50 to about 1:1.

13. A mineral oil composition selected from the group consisting of fuel and lubricant compositions, comprising a major amount of a mineral oil and a minor amount, sufficient to confer corrosion inhibiting properties upon the composition, of a combination of (*a*) mixed oil-soluble n-alkyl amines containing 8 to 18 carbon atoms and (*b*) a substantially neutral addition product of propylene oxide and 3-methylbutyl,2-ethylhexyl acid o-phosphate, where the weight ratio of said amines and said substantially neutral addition product is about 1:50 to about 1:1.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 2,934,500 April 26, 1960

Troy L. Cantrell et al.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, line 10, for "-dimethylonyl" read -- -dimethyl-nonyl --; column 4, line 69, for "to" read -- of --; column 5, line 15, in the table, for "Calonmel" read -- Calomel --.

Signed and sealed this 20th day of September 1960.

(SEAL)

Attest:

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Commissioner of Patents

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