United States Patent Office 3,458,444 Patented July 29, 1969

3,458,444

RUST INHIBITING COMPOSITION

William T. Shepherd, Port Arthur, Howard J. Platte, Groves, and Federico P. Gonzalez, Nederland, Tex., assignors to Texaco Inc., New York, N.Y., a corporation of Delaware

No Drawing. Filed Nov. 17, 1967, Ser. No. 683,774
Int. Cl. C10m 1/32; C23f 11/14
U.S. Cl. 252—51.5 5 Claims

ABSTRACT OF THE DISCLOSURE

Rust and sludge inhibiting additive for mineral lubricating oils comprising the reaction product of an alkenyl succinic acid or anhydride and a tertiary amine represented by the formula R—N—$(R'OH)_2$ in which R is an aryl or alkaryl radical and R' is a divalent alkylene radical having from 2 to 4 carbon atoms and mineral lubricating oil composition containing said rust and sludge inhibitor.

This invention relates to a novel rust inhibiting composition formed by the reaction of an alkenyl succinic acid and/or anhydride with an alkanolamine and to a turbine lubricating oil composition containing the rust inhibitor.

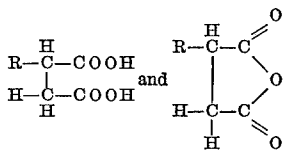
Alkenyl succinic acids and anhydrides have heretofore been reacted with aliphatic amines and with certain alkanolamines in conjunction with aliphatic acids to produce reaction products exhibiting rust inhibiting properties, see U.S. 2,540,800 and U.S. 2,638,449. The level of rust inhibition imparted by these materials, however, has not been entirely satisfactory. In addition, some of the known reaction products have been ineffective for preventing the formation of sludge when employed in a turbine lubricating oil composition. In contrast to the known rust inhibitors, the reaction product of the present invention is highly effective as a rust inhibitor in the ASTM Turbine Oil Oxidation Test (D-943) and, surprisingly, also for reducing or substantially preventing the formation of sludge in a turbine lubricating oil composition.

In accordance with this invention, there is provided a rust inhibitor comprising the reaction product of a reactant, selected from the group consisting of an alkenyl succinic acid and an alkenyl succinic anhydride in which the alkenyl radical has from 8 to 18 carbon atoms, and a tertiary amine compound, represented by the formula:

$$R—N—(R'OH)_2$$

in which R is an aryl or alkaryl radical having from 6 to 10 carbon atoms and R' represents a divalent alkylene radical having from 2 to 4 carbon atoms, in a mole ratio ranging from about 1:1 to 2:1 respectively, and at a temperature from about 80 to about 125° C. There is also provided a rust and sludge inhibited turbine lubricating oil composition comprising a major proportion of mineral lubricating oil with conventional additives and a minor amount of the reaction product defined above.

The alkenyl succinic acid and anhydride components employed in making the additive of the invention are represented by the following formulas:



in which R represents an alkenyl radical having from 8 to 18 carbon atoms. Typical alkenyl succinic acids or

anhydrides which can be employed are dodecenyl succinic acid and anhydride decenyl succinic acid and anhydride, tetradecenyl succinic acid and anhydride and the like.

The tertiary amine component of the reaction product is represented by the formula:

$$R—N—(R'OH)_2$$

in which R is an aryl or alkaryl radical having from 6 to 10 carbon atoms and R' represents a divalent alkylene radical having from 2 to 4 carbon atoms. The presence of an aryl or alkaryl radical on the nitrogen atom in the alkanolamine component of the reaction product appears to be essential to the preparation of an effective rust-inhibiting and sludge-inhibiting reaction product which does not degrade other desired properties possessed by a turbine lubricating oil. For example, reaction products in which R is an alkyl radical on the nitrogen atom have the undesirable property of degrading water separating characteristics of the turbine oil composition.

Tertiary amine compounds which will form effective rust and sludge inhibiting reaction products include: phenyldiethanolamine, tolyldiethanolamine, isopropylphenyl diethanolamine, ethylphenyl diethanolamine, phenyl dipropanolamine and the like.

The rust inhibiting reaction product of the invention is prepared by reacting the alkenyl succinic acid or anhydride and the above-defined tertiary amine in approximately 1:1 to 2:1 mole ratios respectively. The components of the reaction product are admixed either with or without the aid of a mutual solvent and reacted at a temperature from about 80° to about 125° C. A catalyst, such as p-toluene sulfonic acid can be used to promote reaction.

The following examples illustrate the preparation of the reaction product of the invention.

EXAMPLE I

830 grams (4.58 moles) of N-phenyl diethanolamine were dissolved in 2200 ml. of benzene and heated at reflux temperature for about 2 hrs. to remove the water present in the ethanolamine. 1220 grams (4.58 moles) of dodecenyl succinic anhydride were added to the benzene solution over 80 minutes while the solution was maintained at a temperature from 188–204° F. After all of the anhydride had been added, the reaction was continued for about 4 hours at about 200–201° F. The benzene was then removed by distillation and by blowing nitrogen through the reaction product. 2063 grams of the reaction product (Additive A) was recovered having a Neutralization No. of 121 as compared to a calculated Neut. No. of 126.

EXAMPLE II

27.4 grams (0.20 mole) of N-phenyl diethanolamine were dissolved in 300 ml. of benzene and heated at reflux temperature to remove water. 106.4 grams (0.40 mole) of dodecenyl succinic anhydride were added to the benzene solution and the mixture reacted at a temperature from 180 to 184° F. for 2 hours. The benzene was removed by distillation and 135 grams of product recovered (Additive B) having a Neutralization No. of 170 as compared to a calculated Neut. No. of 168.

The lubricating oil composition of the invention comprises a refined mineral lubricating oil containing conventional turbine oil additives and from about 0.01 to 5 weight percent of the reaction product of the invention. The preferred concentration of the reaction product in the lubricating oil is from about 0.025 to 0.5 weight percent. The reaction product is readily soluble in mineral lubricating oils and can be readily blended therein to prepare the lubricating composition.

The rust-inhibiting and sludge-inhibiting properties of the reaction product of the invention were determined in the ASTM Oxidation Test (D–943) wherein the oil sample is subject to a temperature of 95° C. in the presence of water, oxygen and an iron-copper catalyst. This test is particularly designed to determine the oxidation characteristics of steam-turbine oils. The base blends employed in these tests, Blends A through D, were refined paraffin base mineral oils containing 0.30 weight percent ditertiary butyl cresol, 0.033 weight percent tetrapropenyl succinic acid, and 6 p.p.m. silicone polymer, which are widely used in inhibit oxidation, rusting, and foaming, respetively in steam turbine oils. Base Blend E was the same as Base Blend C plus 0.1 weight percent of an alkylated aromatic aimine added as supplemental oxidation inhibitor. These base blends had the following characteristics.

	Base Blend				
	A	B	C	D	E
Gravity, API	30.0–33.0	29.0–32.0	28.5–31.5	27.0–30.0	28.5–31.5
Flash COC, F. (min.)	390	400	410	460	410
Vis.SUS at 100° F	140–150	200–225	300–315	650–700	300–315
Color ASTM (max.)	1.0	1.0	1.0	2.0	1.0
Pour, F. (max.)	0	5	5	5	5
Neut. No. (max.)	0.10	0.10	0.10	0.10	0.10

The Oxidation Test was run until the acidity of the oil sample reached a Neutralization No. of 2.0. The length of time it took to reach this end point is given in hours. Likewise, the time to formation of rust on the steel coils and sludge in the oil is shown in hours. The results are the average for two or more test samples.

ASTM OXIDATION TEST

Base blend	Additive, wt. percent	Hours to—		
		2.0 Neut. No.	Rust	Sludge
Oil A	None	2,681	888	520
Do	0.05 Additive A	3,112	None	None
Do	0.025 Additive B	3,092	None	None
Do	0.05 Additive B	3,221	None	None
Oil B	None	1,718	696	696
Do	0.05 Additive A	2,602	None	None
Oil C	None	1,720	664	1,000
Do	0.025 Additive A	2,108	None	None
Do	0.05 Additive A	2,692	None	None
Do	0.05 Additive B	1,866	None	None
Oil D	None	1,609	444	444
Do	0.05 Additive A	2,383	None	None
Do	0.05 Additive B	2,540	None	None
Oil E	None	2,800	436	720
Do	0.03 Additive A	3,222	None	None
Do	0.025 Additive B	2,940	None	None

The foregoing test results show that the reaction products of the invention very substantially improved the oxidation resistance of the lubricating oil composition. More surprisingly these anti-rust agents completely arrested the formation of rust and the deposition of sludge in the improved lubricating oils.

Obviously numerous modifications and variations of the invention may be made without departing from the spirit and scope thereof, and therefore, only such limitations should be made as are indicated in the appended claims.

We claim:

1. A turbine lubricating oil composition comprising a mapor proportion of a mineral lubricating oil and a minor rust inhibiting amount of the reaction product of a reactant selected from the group consisting of an alkenyl succinic acid and an alkenyl succinic anhydride in which the alkenyl radical has from 8 to 18 carbon atoms, and a tertiary amine reactant represented by the formula:

$$R—N—(R'OH)_2$$

in which R is an aryl or alkaryl radical having from 6 to 10 carbon atoms and R' is a divalent alkylene radical having from 2 to 4 carbon atoms in a mole ratio ranging from about 1:1 to 2:1 respectively and at a temperature ranging from about 80° to about 125° C.

2. A lubricating oil composition according to claim **1** containing from about 0.01 to 5 weight percent of said reaction product.

3. A lubricating oil composition according to claim **1** in which said alkenyl succinic anhydride is dodecenyl succinic anhydride and said reaction is conducted using a mole ratio of about 1:1.

4. A lubricating oil composition according to claim **1** in which said tertiary amine reactant is N-phenyl diethanolamine.

5. A lubricating oil composition according to claim **1** containing from about 0.05 to 1 weight percent of ditertiary butyl cresol and from about 0.01 to 0.15 weight percent of tetrapropenyl succinic acid.

References Cited

UNITED STATES PATENTS

2,588,412	3/1952	Rocchini.
2,604,451	7/1952	Rocchini.
3,183,070	5/1965	Udelhofen.
3,368,971	2/1968	Retzloff et al.

DANIEL WYMAN, Primary Examiner

W. J. SHINE, Assistant Examiner

U.S. Cl. X.R.

252—392, 403