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# COMPOSITIONS OF MATTER HAVING ANTI-RUST PROPERTIES

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This invention relates to novel rust inhibited normally liquid oil compositions and especially petroleum products wherein the composition is in the presence of a rust producing amount of water. More particularly, this invention relates to novel rust inhibitors and their inclusion in normally liquid oils in contact with rust producing water for inhibiting the rusting of ferrous metal in contact with the oil.

The rusting of steel and other ferrous metals used in the transportation and storage of petroleum products has always presented a serious problem in the petroleum industry. The rusting of pipelines and tanks used to transport normally liquid oil products represents in itself a substantial annual economic loss in maintenance and replacement costs. In addition, the presence of sediment and rust as a result of the rusting action of water and carryover of such sediment and rust into fuel burning installations creates fuel quality and operating problems. Such sediment and rust also creates special problems in the case of aviation gasoline where the hazard of engine stoppage through clogging of filters and carburetion equipment presents special hazards. The rusting problem also applies to the handling and use of motor and diesel fuels as well. A particular problem is created by rust formation in lubricating oils such as engine lubricating oils where, during normal lubrication operations, rusting of ferrous metal parts and the presence of resulting rust particles may create uneven contact of moving metal parts with resulting increased friction. In such lubricating operations, surface wear of moving parts is greatly increased.

Further, the rusting of fuel storage tanks used to supply industrial and household burner installations and the rusting of lubricating oil storage tanks used to supply industrial machinery present serious problems. Although the rusting of such storage tanks may cause eventual breakthrough of storage tank walls and result in losses arising from the necessary replacement of storage tanks, the greater problem results from the accumulation of rust in petroleum products and particularly in normally liquid oils and the future use of such oils and products. Thus, clogging of filters, burners, feed lines, etc., as well as undue wear in industrial machinery and internal combustion engines with ultimate destruction of such machinery or engines and their intended use may result.

The problem of rusting is associated with the presence of moisture, i.e., water, in the oil products by entrainment, condensation, and solution. In most cases, the problem is accentuated by the presence of a separate water phase. Thus, in the storage and bulk shipment of products such as gasoline, it is common practice to maintain a water layer as tank bottoms. Even where a water layer is not used as tank bottoms, a separate water phase may form by continual or repeated condensation of moisture, associated with tank breathing or the alternate expansion and contraction of the bulk with temperature changes unless special precautions are taken.

In the particular case of lubricating oils present in internal combustion engines, water may also be accumulated by condensation from air admitted to the crankcase through crankcase breathing, and water may also be admitted into the crankcase by blow-by from the combustion chamber.



Therefore, the problem of rust inhibition of oils in the presence of water is a universal problem applicable to at least some extent to a wide variety of petroleum oils. Thus, it becomes necessary to impart to such oils an ability to protect ferrous metals against rusting normally caused by the presence of water. Many addition agents have been proposed for this purpose. It has been proposed to add to such oil products a rust inhibiting agent which is capable of protecting ferrous metal parts. Many such addition agents have been proposed. In the past, fatty acids have been widely used as rust inhibitors. However, fatty acids have a disadvantage in that they may become corrosive to certain non-ferrous metals. This problem is particularly acute in the case of internal combustion engine lubricating oils wherein operation is at high temperature and non-ferrous alloy bearings are present.

We have now provided rust inhibited oil compositions which protect against rusting of ferrous metals in the presence of normally rust producing amounts of water. The rust inhibiting addition agents of this invention do not contain free fatty acid groups and thereby overcome the difficulty encountered in the use of fatty acid rust inhibitors. Accordingly, our present invention provides a normally liquid oil, such as, for example, a petroleum oil, in the presence of water and containing a rust inhibiting minor amount of an oil-soluble rust inhibiting composition selected from the group consisting of a boric acid complex of a triol monoester, an alkanol ester thereof, a dialkanol amine salt thereof and a dialkyl dithiophosphate thereof. The triol portion of the complex is an open chain hydrocarbon triol group having from 3 to 10 carbon atoms and the monoester group is an open chain hydrocarbon group having an ester linkage with the triol group. The open chain hydrocarbon portion of the monoester group may contain from 5 to 18 carbon atoms. The small amount of such oil-soluble composition contained in the present compositions is an amount sufficient to inhibit rusting normally caused by the water.

Although greater or lesser amounts of the boric acid complex of the triol monoester (referred to hereinafter as the triol monoester-boric acid complex), ester thereof, salt thereof or dialkyl dithiophosphate thereof may be used in the composition of this invention, it is advantageous to use from about 0.001 to about 10 weight percent of the oil-soluble composition based on the normally liquid oil. The preferred range of concentration based on oil is from about 0.001 to about 2.0 weight percent of the oil-soluble composition. Of course, the amounts may be varied as desired and as required for a particular use in rust inhibition. For example, if it is anticipated that the oil product will come into contact with or accumulate greater amounts of water, more of the oil-soluble rust inhibiting composition may advantageously be included therein.

The oil-soluble triol monoester-boric acid complex of this invention is conveniently formed by the reaction of approximately equimolar amounts of a triol monoester and boric acid. The reaction temperature may be any temperature from 0° C. to the decomposition temperature of the reactants. Useable convenient temperatures may be advantageously in the range of from about 50 to about 250° C. Temperatures above about 100° C. are preferred. It is also preferred to carry out the reaction at a temperature below the boiling point of the triol monoester in order to eliminate the necessity for pressurizing the reaction mixture to keep the triol monoester from distilling off. The reaction may conveniently be carried out at the reflux temperature of the reaction mixture using an overhead condenser to condense and return triol monoester to the reaction mixture. Also, the reaction may conveniently be carried out in the presence of

an inert solvent such as xylene, benzene, toluene and the like at the reflux temperature of the solvent.

The reaction is carried out until more than one mole of water per mole of boric acid or per mole of triol monoester is produced as a reaction by-product. The water may conveniently be taken overhead, for example, through a condenser adjusted to return triol monoester or solvent to the reaction mixture, and may be collected and measured to determine the amount of water formed as a by-product. Other means such as liquid traps for collecting and/or measuring water formed during the reaction, are well known to the art. Although the boric acid and triol monoester are reacted in approximately equimolar amounts as set out above, an excess of either reactant may be included in the reaction mixture. Thus, the moles of water are determined with reference to the moles of the reactant present in lesser amounts in the reaction mixture. The desired product is formed after at least one mole of water is split out from the reactants per mole of reactable triol monoester, i.e., reactable with boric acid. Thus, the reaction is continued until at least such one mole of water is formed. The formation of 100% complex is assumed to be theoretically complete when two moles of water are formed. The reaction proceeds readily toward completion but completion to the extent of 100% complex formation is not necessary. The presence of unreacted triol monoester and boric acid in the present compositions is not undesirable and does not adversely affect rust-inhibiting properties. It is only necessary that more than one mole of water be formed. Advantageously the reaction may be permitted to proceed until at least 1.5 moles of water are formed, indicating formation of 50% of the complex in the reaction mixture. Preferably the reaction may be continued to substantial completion, i.e., until about 2 moles of water are formed per mole of reactable triol monoester or per mole of reactant present in the lesser molar amount.

The reaction time is determinable for any combination of reactants by recovering and collecting water formed as a by-product as discussed above. As an example, it has been found that at about 140–200° C. reaction temperature range the reaction is usually sufficiently progressed to form an acceptable rust inhibitor in three hours or less.

As indicated above, molar excesses of either reactant may be included in the reaction mixture since an excess of either reactant in the product will not adversely affect the product or the function of the product. However, any insoluble inorganic boron compounds resulting from the inclusion of an excess of boric acid in the reaction mass may be removed, if desired, such as by filtering.

It is preferred, for complete utilization of the reaction, that the reaction be carried out until about 2 moles of water are formed as a by-product for each mole of triol monoester or boric acid included in the lesser amount in the reaction mixture.

The triol monoesters may conveniently be prepared by reacting equimolar amounts of a triol having 3 to 10 carbon atoms and a carboxylic acid having 5 to 18 carbon atoms with an esterification catalyst under known esterification conditions. The resulting triol monoester, used for complexing with boric acid in accordance herewith, therefore, contains from 3 to 10 carbon atoms in the triol group and 5 to 18 carbon atoms in the monoester group. It is preferred that the triol group and/or the monoester group be sufficiently straight chained to provide the characteristic of oil-solubility to the complex. It is advantageous that the free hydroxy groups of the triol monoester have a chain of 1 to 5 and preferably 2 to 3 carbon atoms between them. Examples of typical triol monoesters useable in forming the complexes of this invention are glyceryl monocaprylate, glyceryl mono-oleate, glyceryl monohexadecanoate, glyceryl monocaprate, glyceryl monomyristate, glyceryl monopalmitate, glyceryl monopentanoate, 2,2-dimethylol-butyl caprylate, 2,2-diethylol-amyl laurate, 2,2-dimethylol-octyl oleate, 2,3-dimethylol-hexyl hexanoate, 1-methyl - 5,5 - dimethylol-hexyl decanoate, 2,2-dimethylol-propyl stearate, 2,2-dimethylol-butyl pentanoate, 2,2-dimethylol-butyl caproate, 2,2-dimethylol-butyl laurate, 9,10-dihydroxy-decyl oleate, 4,5-dihydroxy-hexyl laurate, 6-methylol-7-hydroxyl-heptyl linoleate, 2,2-dimethylol heptyl stearate, etc.

The boric acid of the present complexes may be derived from the inclusion of boric acid or boron compounds, such as boric acid anhydride, capable of forming the boric acid in situ in the reaction mixture. Where boric acid anhydride is used, amounts of water recovered from the complexing reaction should be corrected for water used to hydrolyze the anhydride.

The alkanol ester of the triol monoester-boric acid complex intended for use as a rust inhibiting agent of this invention, i.e., in the presence of water, may be a $C_{1-18}$ alkanol ester of the complex and preferably a $C_{1-12}$ alkanol ester. The ester is formed by reacting the alkanol with the complex under the usual and well known esterification conditions. Acid catalyst may be used if desired. Accordingly, the reaction may conveniently be carried out at elevated temperature with reference to room temperature and the reaction rate may be increased, if desired, raising the reaction temperature, as is known to the art. The esterification reaction is permitted to proceed until one mole of water is split out per mole of ester formed. Water may be recovered and measured in the same manner as in the boric acid reaction. Examples of the useable alkanol esters of triol monoester-boric acid complex are the esters of the following alcohols: methanol, ethanol, propanol, isopropanol, butanol, 2-methyl butanol, hexanol, 2-ethyl hexanol, 2,3-dimethyl hexanol, 1,6-hexane diol, n-octanol, and decanol, n-decenol, n-dodecanol, 7-ethyl decanol, 4,4,8,8-tetramethyl dodecanol, n-hexadecanol, n-octadecanol, n-octadodecenol, neo-hexanol, 2,3,5-trimethyl pentanol, etc., and the triol monoester-boric acid complex.

The dialkanol amine salt of the triol monoester-boric acid complex may be prepared by reaction of a dialkanol amine with the complex. The reaction proceeds readily at room temperature but the reaction rate may be increased, if desired, by raising the reaction temperature above room temperature. The reaction conditions employed are the same reaction conditions normally employed for formation of amine salts. Such conditions are well known to those skilled in the art. The dialkanol amine salts are the di($C_{1-6}$ alkanol) amine salts and preferably the di($C_{2-3}$ alkanol) amine salts of the complex. Examples of such salts are the dimethanol amine, diethanol amine, dipropanol amine, dibutanol amine, dipentanol amine, dihexanol amine, di(2-methyl butanol) amine, diisopropanol amine, di(2,3-dimethyl butanol) amine, etc., salts of the triol monoester-boric acid complex.

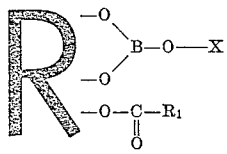
The dialkyl dithiophosphates of the monoester triol-boric acid complex are the di($C_{1-18}$ alkyl) dithiophosphates and may be prepared by reacting the complex with the corresponding dialkyl dithiophosphates and splitting out $H_2S$. The dialkyl dithiophosphate reacts in approximately equimolar amounts with the complex based on boron of the complex. However, it is fully intended that greater or lesser than molar amounts of dialkyl dithiophosphate may be used in the reaction. An excess of dialkyl dithiophosphate may be advantageous as an effective corrosion inhibitor, especially where the resulting product is to be utilized in an engine lubricating oil. Of course, since the complex itself is a rust inhibitor, an excess of the complex is certainly not undesirable in the present compositions. The reaction is apparently a condensation reaction during which hydrogen sulfide is split out and completion of the reaction may be determined by the cessation of evolution of hydrogen sulfide. The same temperatures may be used for the dialkyl dithiophosphate reaction as have been described above for use with the esterification reaction. The preferred temperature range is 100° C. to 200° C. Other temperatures and reaction

conditions, normally used in condensation reactions with resulting splitting out of hydrogen sulfide, may be used in forming the dialkyl dithiophosphates.

Preferred dialkyl dithiophosphates of the complex are the di($C_{3-12}$ alkyl) dithiophosphates. Preferably the alkyl group is a saturated hydrocarbon group. Examples of dialkyl dithiophosphates of the complex are the dimethyl dithiophosphate, diethyl dithiophosphate, dibutyl dithiophosphate, isopropyl hexyl dithiophosphate, isopropyl n-decyl dithiophosphate, di(n-decyl) dithiophosphate, dilauryl dithiophosphate, dihexyl dithiophosphate, di(3,4-dimethyl-hexyl) dithiophosphate, di(2-ethyl-hexyl) dithiophosphate, diisopropyl dithiophosphate, dioleyl dithiophosphate, pentyl n-decyl dithiophosphate, 4,6-dipropyl-n-decyl methyl dithiophosphate, di-n-octadecyl dithiophosphate, di-n-hexadecyl dithiophosphate, ethyl n-heptadecyl dithiophosphate, di(3,3,6,6-tetramethyl-dodecyl) dithiophosphate and methyl octadecyl dithiophosphate of the triol monoester-boric acid complex. Other useable dialkyl dithiophosphates of the complex will be evident to those skilled in the art.

The rust inhibiting compositions of this invention are soluble in the oil product in which they are included and, therefore, are defined as "oil-soluble." Amounts exceeding the solubility of the complex in oil are neither necessary nor desired.

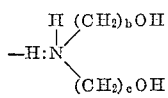
The splitting out of water as a by-product in formation of the boric acid-triol monoester complex of this invention indicates ester formation. Thus, it is believed that at least some esters form during the reaction and, although the structure of the esters is unknown, it is believed that the rust inhibiting compositions of the present invention include at least small amounts of compositions having the structural formula:



and polyacid derivatives thereof. In the above formula, R is an open chain (non-cyclic) hydrocarbon group having from 3 to about 10 carbon atoms; $R_1$ is an open chain hydrocarbon group having from about 5 to about 18 carbon atoms and preferably from 7 to 12 carbon atoms; X is selected from the class consisting of hydrogen, a hydrocarbon alkyl group having from 1 to about 18 carbon atoms, a thiophosphate group corresponding to the structural formula:



and a dialkanol amine salt group corresponding to the structural formula:



$R_2$ and $R_3$ are each selected from the class consisting of hydrogen and a hydrocarbon alkyl group having from 1 to about 18 and preferably from 3 to 12 carbon atoms; and *b* and *c* are each integers of 1 to 6 inclusive and preferably 2 to 3 inclusive. R, $R_1$, $R_2$, and $R_3$, may be saturated or unsaturated hydrocarbon groups but are preferably saturated. R, $R_1$, $R_2$, and/or $R_3$ are sufficiently straight chain to provide the characteristic of oil solubility to the triol monoester-boric acid complex.

The above formula may appear to be correct with reference to prior art theories such as that of Thomas et al., U.S. 2,795,548, patented June 11, 1957. However, we have been unable to substantiate the above formula of the complexes of this invention and it is not intended that this



invention be limited to any theory concerning the structure of the complexes.

The normally liquid oils of the compositions of this invention are those normally liquid oils boiling in the gasoline through lubricating oil boiling range, and especially petroleum oil products boiling in that range. Broadly, such oils include gasoline, other hydrocarbon fuel oils, and lubricating oils. The oils may be gasoline blending stocks such as reformates and virgin and cracked naphthas or the oil may be a blended gasoline stock. The lighter oils useable include gasoline and the hydrocarbon fuel oils normally used in burner installations, diesel engines, as well as those used for insecticide carriers. For example, the oils of the composition of this invention may be residual or distillate fuel oils such as diesel fuel, jet fuel, heavy industrial residual fuel (e.g., bunker C), furnace oil, heater oil fractions, kerosene, gas oil, etc. Of course, any mixtures of residual and/or distillate oils are also intended. The fuel oil may be a virgin or cracked petroleum distillate fuel oil and may advantageously boil in the range of from about 200 to about 750° F. Such fuel oils may contain or consist of cracked components, such as for example, those derived from cycle oils or cycle oil cuts boiling in the gasoline range or heavier and usually boiling in the range of from about 450 to about 750° F. Such cycle oils may be derived by catalytic or thermal cracking. High sulfur fuels and low sulfur fuels such as diesel oils and the like may also be used. The distillate fuel oil is preferably a hydrocarbon distillate fuel oil.

Also intended are the oils used for lubricating purposes and/or metal working purposes. Such oils include the cutting oils, drawing oils, grinding oils and other extreme pressure oils. Also intended are the glass grinding oils which are usually blends of kerosene containing dispersance addition agents. Other useable lubricating oils are those normally falling in the lubricating viscosity range. Such lubricating oils include the hydrocarbon or mineral lubricating oils of naphthenic and/or paraffinic types. Such lubricating oils may be derived by conventional methods such as solvent extraction and acid treatment. Synthetic oils may also be used. Such synthetic oils include the synthetic hydrocarbon oils of the alkylene polymer type, the alkylene oxide polymers and their derivatives such as propylene oxide polymers and esters thereof, dicarboxylic ester synthetic oils including dibutyl adipate, diethyl hexyl sebacate, fumarate polymers, dialkyl azelates, pelargonates, etc. Other synthetic lubricating oils which are useable are the alkyl benzene oils such as tetradecyl benzene. The non-hydrocarbon synthetic lubricating oils are also intended; such oils include the polysiloxanes such as polyalkyl, polyaryl, polyalkoxy, etc., siloxanes and the silicate ester oils such as the tetraalkyl silicates. The hydrocarbon oils are preferred.

As indicated above, other addition agents such as dispersancy agents and extreme pressure agents may be included in the liquid oil. Such other addition agents are used to perform particular functions. Thus, where desired, pour point depressants, corrosion inhibitors, combustion improvers, viscosity index improvers, oiliness agents, and the like may also be added.

The following examples illustrate the compositions of the present invention and are included for the purpose of illustration and description of the invention, and not for purposes of limitation of the scope of the invention.

## EXAMPLE I

This example illustrates the preparation of a triol monoester-boric acid complex useable in our present invention. In this example, trimethylolpropane monocaprylate borate was prepared by reacting boric acid with trimethylolpropane monocaprylate. The trimethylolpropane monocaprylate was prepared by reacting equimolar amounts of trimethylolpropane and octanoic acid in xylene solvent at a temperature of about 145–150° C. until about 1 mole of water per mole of trimethylolpropane was

taken overhead and collected. The resulting 386 grams (1.43 moles) of trimethylolpropane monocaprylate were mixed with 88 grams (1.43 moles) of boric acid in 100 ml. of xylene solvent and the resulting mixture was heated at about 140° C. for 2½ hours. During the 2½ hours of heating, 53 ml. of water were taken overhead and collected. Thereafter, the heating was discontinued and the reaction mixture was permitted to cool to about room temperature (25° C.) The reaction mixture was then filtered through Celite (diatomaceous earth). The filtered product had an acidity of 169 mg. KOH/gm. and contained 1.36% boron.

## EXAMPLE II

The example illustrates the preparation of a dialkanol amine salt of a triol monoester-boric acid complex, which salt is a useful addition agent of this invention. Accordingly, 89 grams (0.3 mole) of the trimethylolpropane monocaprylate borate prepared as in Example I were mixed with 31.5 grams (0.3 mole) of diethanol amine and the resulting mixture was stirred at room temperature (25° C.) for one hour. A white, thick slurry was formed and the reaction mixture was allowed to stand at room temperature until it solidified to a solid white mass. 120 grams of SAE 5 mineral lubricating oil were added and the diluted mixture was stirred. Excess mineral oil was then decanted off and the resulting thick slurry was recovered as the product. The product was the dialkanol amine trimethylolpropane monocaprylate-boric acid complex and contained 0.88% boron and 2.12% nitrogen.

## EXAMPLE III

This example illustrates preparation of another triol monoester-boric acid complex of this invention. Accordingly, glyceryl monocaprylate-boric acid complex was prepared by reacting glyceryl monocaprylate with boric acid. The glyceryl monocaprylate was prepared by reacting equimolar amounts of glycerol and octanoic acid in xylene solvent at 145–150° C. until about 1 mole of water per mole of octanoic acid was removed and collected. 327 grams (1.5 moles) of the glyceryl monocaprylate were mixed with 93 grams (1.5 moles) of boric acid and the resulting mixture was heated for three hours. During the 3 hours, 52 ml. of water were taken overhead and collected. The product was then filtered through Celite. The filtered product contained 2.15% boron and had an acidity of 225 mg. KOH/gm.

## EXAMPLE IV

Still another triol monoester-boric acid complex is illustrated by the preparation of the example. As a starting material a glyceryl monoester (derived from mixed fatty acids predominating in oleic acid and hereinafter referred to as "monooleate") was prepared by reacting equimolar amounts of glycerol (as crude 88% saponifiable glycerine) and Century fatty acid 1475 (a crude oleic acid consisting of mixed fatty acids derived from vegetable oils and predominating in oleic acid as marketed by Harchem Division, Wallace and Tiernan, Inc.), at about 140–200° C. until one mole of water was taken overhead and collected per mole of fatty acid or glycerol (about 6 hours). 1168 grams (4 moles) of the resulting glyceryl "monooleate" was mixed with 248 grams (4 moles) of boric acid and heated at temperatures ranging from 140–200° C. while stirring until 144 ml. of water were taken overhead and recovered. The reaction mixture was then cooled to about room temperature (25° C.) and filtered through Celite. The resulting product was glyceryl "monooleate"-boric acid complex and contained 1.39% boron.

## EXAMPLE V

In this example another glyceryl monooleate-boric acid complex was prepared. Accordingly, 1127 grams (3.16 moles) of a commercial glycerol monooleate (Emery 2221) were mixed with 196 grams (3.16 moles) of boric acid and the resulting mixture was stirred and heated at temperatures ranging from 140–200° C. until 60 ml. of water were taken overhead and collected. The reaction mixture was then cooled and filtered through Celite yielding a clear orange filtrate as a product. The product contained 0.60% boron.

## EXAMPLE VI

The example illustrates the preparation of still another addition agent useful in accordance with this invention. Accordingly, the diethanol amine salt of a triol monoester-boric acid complex was prepared. 60.4 grams (0.2 mole) of a glyceryl "monooleate"-boric acid complex prepared as in Example IV and 21.0 grams (0.2 mole) of diethanol amine were mixed and stirred at room temperature (about 25° C.). The reaction was exothermic and the temperature increased above room temperature. After about 60 minutes, the reaction was complete. The resulting product, i.e., the diethanol amine salt of glyceryl "monooleate"-boric acid complex contained 0.97% boron and 3.53% nitrogen.

## EXAMPLE VII

As another example of preparation of a dialkanol amine salt, 73.2 grams (0.3 mole) of glyceryl monocaprylate-boric acid complex (as prepared in Example III) were mixed with 31.5 grams (0.3 mole) of diethanol amine and the mixture was stirred at room temperature for one hour. A thick, white slurry was formed and the slurry was permitted to stand at room temperature until it solidified into a solid white mass. The mixture was decanted leaving the solid white mass and 84 grams of solvent extract SAE 5 mineral oil were added and stirred. Excess mineral oil was decanted off leaving a thick viscous slurry as a product. The product was the diethanol amine salt of glyceryl monooleate-boric acid complex and contained 1.00% boron and 3.49% nitrogen.

## EXAMPLE VIII

This example illustrates the preparation of a dialkyl dithiophosphate of a triol monoester-boric acid complex. Accordingly, 484 grams (one mole) of a glyceryl monooleate-boric acid complex prepared as in Example IV were mixed with 400 grams (one mole) of di-n-decyl dithiophosphoric acid and the resulting mixture was heated at temperatures of 110–155° C. until $H_2S$ evolution ceased (3 hours and 45 minutes). An attempt was made to filter the cooled reaction mixture through a heated Celite pad but the reaction mixture would not filter. The reaction mixture was then placed in a beaker, stirred and blown with nitrogen to remove $H_2S$. The product was the di-n-decyl dithiophosphate of glycerol monooleate-boric acid complex. The product contained 0.85% boron, 2.2% phosphorous and 4.38% sulfur.

## EXAMPLE IX

This example illustrates the preparation of an alkanol ester of a triol monoester-boric acid complex useful as an addition agent in this invention. Accordingly, 396 grams (1.40 moles) of a glyceryl "monooleate" borate prepared as in Example IV and 135 grams (1.40 moles) of octyl alcohol were mixed and stirred while heating to 200° C. 9.5 ml. of water were taken overhead and collected and the product was then permitted to cool. Although it was calculated that 19.0 ml. of water would be taken overhead during heating, only half that amount was actually obtained, indicating incomplete esterification and presence of unreacted alcohol and glyceryl "monooleate"-boric acid complex in the product. The product contained about 50% of the octyl alcohol ester of glyceryl "monooleate"-boric acid complex and had a boron content of 0.40%.

Samples of addition agents of this invention were tested for their ability to aid in preventing the rusting of ferrous metal parts in the presence of water. Accordingly, tests were conducted in accordance with the ASTM D–655 rust test. Briefly the test procedure involved stirring a mixture

of 300 ml. of each sample (including addition agent and base oil) with 30 ml. of distilled water in a beaker at a temperature of about 140° F. with a cylindrical steel specimen completely immersed therein. The test was continued for a period of 24 hours. At the end of the 24-hour test period, the steel specimens were inspected for rust and rated as to the amount of rust present. A perfect rating indicates no rust present. The samples tested are described in the following table as to the identity of the addition agent and the concentration of the addition agent in the base oil. In all cases the base oil was a white refined mineral oil having a viscosity of 140–145 sec. at 100° C. The results of the rust tests are also reported in the table:

*Rust Test Results*

| Identity of Addition Agent | Concentration in Base Oil, Wt. Percent | Rust Rating |
|---|---|---|
| Example I | 0.5 | Perfect. |
| Do | 0.4 | Do. |
| Do | 0.3 | Do. |
| Do | 0.2 | Do. |
| Do | 0.1 | Trace of Rust (Very slight Rusting). |
| Example II | 0.5 | Perfect. |
| Do | 0.1 | Trace of Rust. |
| Example III | 0.1 | Perfect. |
| Example IV | 0.5 | Do. |
| Do | 0.1 | Do. |
| Do | 0.01 | Do. |
| Example VI | 0.5 | Do. |
| Do | 0.1 | Do. |
| Do | 0.01 | Moderate Rusting. |
| Example VII | 0.5 | Perfect. |
| Do | 0.1 | Do. |
| Do | 0.01 | Moderate Rusting. |
| Example VIII | 0.5 | Perfect. |
| Do | 0.1 | Do. |
| Example IX | 0.5 | Do. |
| Do | 0.1 | Do. |
| None | None | Severe Rusting. |

The above test results demonstrate the utility of the present compositions as rust inhibitors under the severe conditions of the ASTM rust test. Under normal storage conditions, much lesser amounts of the addition agents may be used and the amount may conveniently be varied with the severities of the rust-producing environment as pointed out above.

It is evident from the foregoing that we have provided rust inhibited oil compositions containing the rust inhibitor described hereinabove, effective in the presence of a normally rust producing amount of water.

We claim:

1. A rust inhibited oil composition comprising a major proportion of a normally liquid hydrocarbon oil and a small amount, sufficient to inhibit rusting, of an oil-soluble rust inhibiting composition comprising a $C_1$ to $C_{18}$ alkanol ester of a triol monoester of a boric acid complex wherein the triol group is an open chain hydrocarbon triol having from 3 to 10 carbon atoms and the monoester group is the remainder of an esterified open chain carboxylic acid having 5 to 18 carbon atoms.

2. A rust inhibited composition comprising a major proportion of a normally liquid hydrocarbon oil and a small amount, sufficient to inhibit rusting, of an oil-soluble rust inhibiting composition comprising a di($C_1$ to $C_{18}$ alkyl) dithiophosphate of a triol monoester-boric acid complex wherein the triol group is an open chain hydrocarbon triol having from 3 to 10 carbon atoms and the monoester group is the remainder of an esterified open chain carboxylic acid having from 5 to 18 carbon atoms.

3. The composition of claim 1 wherein said small amount is in the range of from about 0.001 to about 10 weight percent based on said normally liquid hydrocarbon oil.

4. The composition of claim 2 wherein said small amount is in the range of from about 0.001 to about 10 weight percent based on said normally liquid hydrocarbon oil.

5. A lubricant composition comprising a major amount of a base lubricant and an additive amount, effective to inhibit rusting of an oil soluble $C_1$ to $C_{18}$ alkanol ester of a triol monoester of a boric acid complex wherein the triol group is an open chain hydrocarbon triol having from 3 to 10 carbon atoms and the monoester is the remainder of an esterified open chain carboxylic acid having 5 to 18 carbon atoms.

6. The composition of claim 5, wherein said additive amount is in the range from about 0.001 to about 10 weight percent, based on said base lubricant.

7. The composition of claim 6, wherein said additive amount is in the range of from about 0.001 to about 10 weight percent, based on said base lubricant.

8. A lubricant composition comprising a major amount of a lubricant and an additive amount, effective to inhibit rusting of an oil soluble di($C_1$ to $C_{18}$ alkyl) dithiophosphate of a triol monoester-boric acid complex wherein the triol group is an open chain hydrocarbon triol having from 3 to 10 carbon atoms and the monoester is the remainder of an esterified open chain carboxylic acid having from 5 to 18 carbon atoms.

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