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2,413,718

LUBRICANT

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9 Claims. (Cl. 252—49.7)

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This invention relates to addition agents and lubricating compositions containing the same and is a continuation-in-part of our copending application, Serial No. 330,950, filed April 22, 1940.

Present-day mechanical devices require lubricating oils of high film strength and of high oiliness characteristics. It has been found that the present-day hydrocarbon lubricants of the very highest quality are deficient in these two very important characteristics. These two properties are of vital importance under conditions of thin film lubrication where the lubricant has been squeezed from between the friction surfaces due to high pressures, slow speeds, and other causes. It is readily seen that the viscosity or the body of the lubricant plays no part in this type of lubrication and that the remaining film of oil should tend to keep the coefficient of friction as low as possible and must have a very high film strength and be of high oiliness value to prevent rupture of the film of the lubricant, which would cause seizure.

Mechanical devices are being designed for higher pressure operation, and the film strength of the best quality straight hydrocarbon lubricants has been found to be too low and not sufficient; therefore an invention which provides the means of improving the film strength of these lubricants is of great importance to the art of lubricant manufacture and to the designer and fabricator of mechanical devices.

Substantially all machines operate in part or at times totally under conditions of boundary or thin film lubrication, under which conditions the oiliness or unctuosity of the lubricant is the first and primary requisite of efficient operation. Those skilled in the art of lubricant manufacture or machine manufacture will readily appreciate the value of an invention that will improve the oiliness of these otherwise high quality lubricants. Under some circumstances, a very small amount of an addition agent will stabilize a hydrocarbon oil against formation of corrosive oxidation compounds or sludge-forming compounds, and as little as 0.001% is often sufficient.

In starting idle mechanical equipment which is lubricated from a sump by pumping or circulating the lubricant, there is always a short period of time during which the rubbing surfaces must operate under conditions of dry friction if ordinary hydrocarbon lubricants are used. With dry friction, the wear on friction surfaces is extreme; and during cold weather when the lubricant is sluggish or during periods when the lubricating system is not functioning properly for one reason or another, rubbing surfaces may not only suffer considerable wear but may be damaged to the point where they must be replaced. The product of our invention has the very important property of reacting with the metal surfaces, penetrating or adsorbing on the metal, and leaving a film of lubricant with high oiliness character which remains on the metal surface irrespective of the length of time the machine is idle.

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This high oiliness film is very even and results in smooth operation which may be easily discerned by the experienced operator and lubricating engineer.

When the hydrocarbon lubricants are diluted with unburned fuel or with other light hydrocarbons, the small degree of oiliness of the original hydrocarbon lubricant is greatly decreased. We have found that the addition of the product of our invention to hydrocarbon lubricants more than compensates for the loss in oiliness and load-carrying ability by dilution.

One object of our invention is to provide a lubricating composition having improved film strength and extreme pressure characteristics.

Other and further objects of our invention will appear from the following description.

We have found that certain compounds are adsorbed or absorbed by metals forming a plurality of layers or films on the surface of the metal, which films are very tenacious (they have a high film strength) and have the property of reducing the coefficient of friction to a greater extent than any known lubricant. The compounds which most readily are adsorbed by metals are ones consisting of polar molecules, that is, molecules of unsymmetrical electrical character which contain an atom or group of atoms exhibiting a secondary or residual valence. These polar compounds tend to produce regimentation of the molecules of hydrocarbon oil when added thereto. If a metal is immersed in a strongly polar compound, a regimentation or cybotaxis of the molecules will take place in the adsorbed film, the molecules being oriented with respect to the surface of the metal by which they are adsorbed. The films of polar compounds, adsorbed by a metal, can be plainly seen and the orientation of the polar molecules on the metal surface ascertained by X-ray diffraction methods, in which a monochromatic X-ray beam is passed through the adsorbed films at an angle. The series of films acts as a diffraction grating, and a diffracted beam will be reflected from the planes of the films and can be recorded photographically. The X-ray photographs will clearly show the orientation of the molecules by characteristic pictures.

X-ray investigation has shown that strongly polar molecules form adsorbed films of unusual thickness and regularity of arrangement. Thicknesses of films of 9,000 Angstrom units have been obtained, which are of enormous magnitude when compared with molecular dimensions.

Broadly stated, our invention comprises the making available for use as addition agents in lubricating compositions of halogen bearing or-

gano-boron esters of the type which contain the >B—OR group (in which R is an organic radicle) and improved lubricating compositions containing minor amounts of such addition agents.

The use of organo-boron compounds broadly in lubricating compositions is not new. Their principal use in lubricating compositions is disclosed in Patent No. 2,234,581, wherein acidic organo-boron compounds are disclosed for the purpose of preventing oxidation of lubricants, especially when the same are subjected to high temperatures and gases which tend to promote oxidation. That patent, as indicated, relates to the use of acidic materials, and the amounts specified for use are in all cases "minute." While organic-substituted boric acids, that is, compounds having the >B—OH group, are disclosed therein, these are described as being of low solubility and less desirable.

In addition to the disadvantage of low oil-solubility, the acidic organo-boron compounds are corrosive to sensitive bearing metals, such as are commonly used in modern internal combustion engines.

We have found, however, that a much more satisfactory sub-generic class of organo-boron compounds for the foregoing uses are those of the ester type and which are additionally characterized by the presence of combined halogen in the molecule.

The ester type material is preferable for use in that it does not have the objectionable high reactivity above pointed out which is possessed by the acidic material. In the same concentrations as suggested by the prior art for the acidic type material, our ester type material is quite effective from the standpoint of inhibiting oxidation without, however, possessing the undesirable properties of the acidic material. The additional characteristic of our improved addition agent, namely, that it contains halogen, gives rise to further advantages, the most important of which may be enumerated as follows:

The presence of halogen in the molecule renders the compounds much more strongly polar, and accordingly they are attracted to the metallic surfaces with which the lubricant comes in contact and which the lubricant is designed to protect from scuffing and scoring, due to overload and the like. This strongly polar nature of the compounds makes it possible to achieve effects on the metal surfaces, such as the bearings, which are not only impossible by the prior art uses of the acidic materials, but which the prior art deliberately sought to avoid by keeping down to as low as possible the concentrations within which such acidic materials were employed.

The addition agents of our invention are desirably strongly polar for the reason that one of the principal purposes for which they are present in the lubricating compositions of our invention is dependent upon their ability to react in a chemical or physico-chemical fashion with the bearing surfaces being lubricated. This chemical or physico-chemical action by which the addition agents of our invention improves the lubricating compositions containing the same is two-fold, and the effects thereof are likewise two-fold.

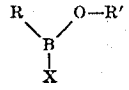
In the first place, the strongly polar nature of the addition agents causes the same to be oriented as an adsorbed film on the bearing surfaces sought to be lubricated with the attended advantages, such as higher "oiliness"; less tendency of the lubricant to drain from the bearing surfaces, etc., all as previously explained.

A second and equally important effect of the increased polarity of our improved addition agents is the increased film strength of the lubricant containing the same, attributable to the function of the cooperative effect of the halogen and the R>BOR' group present in the molecule as a film strength improver.

The presence of halogen in the molecule has an additional advantage in that it renders the compound much less volatile so that it will not be so readily evaporated from the lubricating compositions during use at high temperatures. At this point it may be well to note that for most purposes the addition agents should have a vapor pressure less than atmospheric at a temperature of 140° C. and preferably at 170° C.

When one of the desired effects of the presence of the addition agent is an increase in the film strength of the lubricant, the ester materials of this invention are particularly advantageous because their higher solubility, as compared with the acidic materials of the prior art, makes it possible to incorporate amounts which are much more effective for that purpose; that is, in amounts higher than .75% or preferably above 1.0%. The upper limit of the concentration of addition agents is determined by factors such as the limit of their solubility; reasons of economy, etc. Our invention, therefore, contemplates that these addition agents may be used in lubricating compositions such as those which comprise a mineral oil base in effective amounts up to the limits of their solubility in the oil, there being generally no reason, however, for including more than about 20%.

The broad class of organo-boron esters contemplated for use by this invention contains two important sub-divisions respectively represented by the following formulas:



in which R and R' are organic radicles and X is a radicle of the class consisting of —R'' and —O—R''' where R'' and R''' are organic radicles; and

$$R^d{=}B{-}OR$$

in which R^d is a divalent organic radicle, and R is an organic radicle.

Other sub-divisions of the broad class of esters contemplated hereby are the following:

R_2BOR', in which R and R' are organic radicles, at least one of which contains four or more carbon atoms. An example of this type is chlor diethyl boric acid lauryl ester.

$RB(OR')_2$, in which R and R' are organic radicles, at least one of which contains four or more carbon atoms. An example of this type is chlor phenyl boric acid dilauryl ester.

These two types of organo-boron esters have the general formula $R_nB(OR')_{3-n}$ where n is an integer less than 3, the valence of boron. They contain at least one carbon-boron bond, and at least one organic radicle is removable by saponification. R and R' are usually hydrocarbon radicles but are not necessarily hydrocarbon radicles, and they may contain besides carbon and hydrogen also other elements including oxygen, sulphur, halogen, phosphorus, or metals. Other examples of our compounds are chlorophenylboric

acid dibutyl ester, chlor diamylboric acid cyclohexyl ester, and the like.

Examples of the organic radicles represented by R, R', R'', R''' and R^d, referred to throughout all of the foregoing description, are the following:

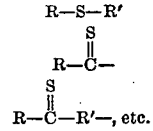
A. Hydrocarbon radicles
1. Alkyl radicles
Amyl (n, iso, sec. and tertiary)
Octyl
Lauryl
Cetyl
Carnaubyl
Ceryl
Montanyl
Mellisyl
Myricyl
Laceryl
Psyllicyl
2. Aryl radicles
Phenyl
Xenyl
Naphthyl (alpha- and beta-)
Anthracyl
Phenanthryl
3. Cyclo-alkyl radicles
Cyclopentyl
Cyclohexyl
Cycloheptyl
Cyclohexyl-cyclohexyl
4. Alkylated aryl radicles
Radicles formed by the alkylation of aryl radicles, such as those listed above (2), with any of the alkyl radicles, e. g., those listed above (1), or with any of the following:
Methyl
Ethyl
Propyl (n- and iso-)
Butyl (n-, iso- and sec-)
Specific examples of alkylated aryl radicles are as follows:
Cresyl
Xylyl
Trimethyl-phenyl
Ethyl-phenyl
Tetra-ethylphenyl
t-Amyl-phenyl
Lauryl-phenyl
Cetyl-phenyl
Ceryl-phenyl
Methyl-naphthyl
Iso-propyl-naphthyl
t-Amyl-naphthyl
Lauryl-naphthyl
Cetyl-naphthyl
Iso-propyl-xenyl
t-Amyl-xenyl
Lauryl-xenyl
5. Alkylated cyclo-alkyl radicles
Radicles formed by the alkylation of cycloalkyl radicles, such as those listed above (3), with any of the alkyl radicles, such as those listed above.
Specific examples are:
Methyl-cyclohexyl
Dimethyl cyclohexyl
t-Amyl cyclohexyl
Lauryl cyclohexyl
Cetyl cyclohexyl
Menthyl
Fenchyl
Bornyl
6. Cyclo-alkylated aryl radicles, e. g.
Cyclohexyl-phenyl
Cyclohexyl-xenyl
Cyclohexyl-naphthyl
7. Arylated-alkyl radicles, e. g.
Benzyl
Phenyl-ethyl
Phenyl-amyl
Phenyl-lauryl
Phenyl-cetyl
Xenyl-methyl
Xenyl-ethyl
Naphthyl-methyl
Naphthyl-ethyl
8. Cyclo-alkylated alkyl radicles, e. g.
Naphthenyl
Hexahydrobenzyl
Cyclohexyl-ethyl
Cyclohexyl-amyl
9. Arylated cyclo-alkyl radicles, e. g.
Phenyl-cyclohexyl
Xenyl-cyclohexyl
Naphthyl-cyclohexyl
10. Mixed radicles, e. g.
Methylcyclohexyl-phenyl
Amylcyclohexyl-phenyl
Cyclohexyl-cresyl
Cyclohexyl-xylyl
Cyclohexyl amyl-phenyl
Cyclohexyl-benzyl
Xylyl-cyclohexyl
Lauryl-phenyl cyclohexyl
Cetyl-phenyl cyclohexyl
Phenyl-methylcyclohexyl
Phenyl-amylcyclohexyl
Xylyl-methylcyclohexyl
Benzyl-cyclohexyl
Benzyl-methylcyclohexyl
Methylcyclohexyl-amyl
Methylcyclohexyl-lauryl

A. Hydrocarbon radicles—Continued.
11. Divalent hydrocarbon radicles
Methylene
Ethylene
Ethylidene
Vinylene
Vinylidene
Propylene
Propylidene
Isopropylidene
Propenylidene
Butylene
Butylidene
Isobutylidene
Amylene
Amylidene
Iso-amylidene
Cyclohexylene
Cyclohexylidene
Phenylene
Phenylidene
Benzal
Cinnamal
Styrolene
Naphthylene
Naphthylidene
Naphthal
Tolylene
Xylylene
Biphenylene
12. Unsaturated hydrocarbon radicles
Vinyl
Allyl
Propenyl (n, and iso-)
Butenyl (n-, and iso-)
Oleyl
Cyclohexenyl
Styryl
Cinnamyl

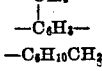
B. Oxygen-containing organic radicles
1. Of the ether type, e. g.
Methoxy-phenyl (anisyl)
Anisal
Ethoxy-phenyl (phenetyl)
Lauroxy-phenyl
Cyclohexoxy-phenyl
Phenoxy-phenyl
Phenoxy-naphthyl
Phenoxy-cyclohexyl
Phenoxy-ethyl
Phenoxy-lauryl
Phenoxy-cetyl
Amyl-phenoxy-ethyl
Phenyl-ethoxy-ethyl
$-C_2H_4-O-C_2H_4-$ (derived from di-ethylene glycol)
$-C_6H_4-O-C_6H_4-$ (derived from diphenyl ether)
Furyl
Fural
2. Of the ketone type, e. g.
Acetyl
Acetyl-phenyl
Acetyl-xenyl
Acetyl-naphthyl
Acetyl-lauryl
Acetyl-cetyl
Acetyl-cyclohexyl
Acetyl-naphthenyl
Crotonyl
Phenacyl
Phenacylidene
Lauroyl
Lauroyl-phenyl
Benzoyl
Benzoyl-phenyl
Benzoyl-naphthyl
Benzoyl-cyclohexyl
Benzoyl-naphthenyl
Benzoyl-lauryl
Benzoyl-cetyl
Stearoyl
Stearoyl-phenyl
Stearoyl-xenyl
Stearoyl-naphthyl
Stearoyl-amyl
Stearoyl-lauryl
Stearoyl-cetyl
Stearoyl-phenoxyphenyl
Stearoyl-cyclohexyl
Stearoyl-naphthenyl
Hexahydrobenzoyl
Naphthenoyl
Naphthenoyl-phenyl
Naphthenoyl-xenyl
Naphtnenoyl-naphthyl
Naphthenoyl-amyl
Naphthenoyl-lauryl
Naphthenoyl-cetyl
Naphthenoyl-phenoxy-phenyl
Naphthenoyl-cyclohexyl
Phenoxybenzoyl-phenyl
Lauryl-benzoyl
Cetyl-benzoyl
Anisoyl
Cinnamoyl
Naphthoyl
Salicylyl
Toluyl
Xyloyl
Furoyl
$-C_6H_4-CO-C_6H_4-$ (derived from salicyl-phenol)
Acetonyl
Acetonylidene
Acrylyl

B. Oxygen-containing organic radicles—Continued.
2. Of the ketone type—Continued.
Adipyl
Benzoylene
Butyryl
Isobutyryl
Carbethoxy
Cresotyl
Desyl
Glutaryl
Glycolyl
Melonyl
Oxalyl
Phthalyl
Succinyl
3. Of the ester type, i. e.
Derived from hydroxy-esters by the removal of the hydroxyl group, as for example, by the removal of the hydroxyl group from the following esters:
a. Esters of hydroxy acids, e. g.
Esters formed by substitution of any of the "R" groups listed above for one or more of the acid hydrogen atoms of the following acids:
Lactic acid
Hydracrylic acid
Citric acid
Tartaric acid
Ricinoleic acid
Hydroxy-stearic acid
Hydroxy-chlorstearic acid
Cyclohexanol carboxylic acids
Hydroxybenzoic acids
Salicylic acid
Hydroxy-phthalic acids
Hydroxy phenyl-benzoic acids
Naphthol carboxylic acids
Esters of polyhydroxy alcohols and phenols in which at least one hydroxyl group remains unsubstituted, for example—esters formed by esterifying the following so that at least one hydroxyl group remains in the resulting ester:
Ethylene glycol
Diethylene glycol
Propylene gylcols
Butylene glycols
Poly-hydroxy benzenes
Dihydroxy-benzenes
Resorcinol
Catechol
Hydroquinone
Tri-hydroxybenzenes
Pyrogallol
4. Hydroxy radicles
Hydroxy-ethyl
Hydroxy-butyl
Hydroxy-amyl
Hydroxy-phenyl
Hydroxy-benzyl
Hydroxy-cyclohexyl
Amyl hydroxy-phenyl
Lauryl hydroxy-phenyl
Cetyl hydroxy-phenyl
Di-amyl hydroxy-phenyl
Lauryl hydroxy-benzyl
Amyl hydroxy-cyclohexyl
Phenyl hydroxy-phenyl
Phenyl hydroxy-benzyl
Hydroxyphenyl-phenyl
Hydroxyphenyl-benzyl
Hydroxybenzyl-phenyl
Hydroxybenzoyl-phenyl
Hydroxy-benzoyl
Hydroxy-stearoyl
Salicylal

C. Sulphur-containing organic radicals.
Examples are any of the radicles listed under B in which a sulphur atom has been substituted for one or more oxygen atoms, e. g.,



where R is an aliphatic radicle (e. g., methyl, ethyl, butyl, lauryl), a cyclo-aliphatic radicle (e. g., cyclohexyl, methyl-cyclohexyl) or an aromatic radicle (e. g., phenyl, benzyl, tolyl, xenyl, naphthyl), and R′ is a divalent organic radicle, e. g., methylene, ethylene, amylene, phenylene, $-C_6H_4-CH_2-$, $-C_6H_4-C_6H_4$



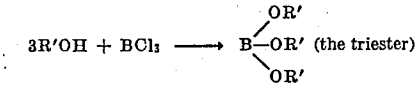
hexahydrophenylene, $-C_6H_{10}CH_2-$, $-C_6H_{10}-C_6H_{10}-$, etc.
Specific examples are:
Thiomethoxy-phenyl ($CH_3-S-C_6H_4-$)
Thiophenoxy-ethyl ($C_6H_4-S-C_2H_4-$)
Thiocyclohexoxy-phenyl ($C_6H_{11}-S-C_6H_4-$)
Thio-acetyl
Thio-stearoyl
Thio-naphthenoyl
Thio-benzoyl
Thio-benzoyl-phenyl
Thio-benzoyl-benzyl
Thio-stearoyl-benzyl
Thio-stearoyl-phenyl
Thio-acetyl-phenyl

C. Sulphur-containing organic radicals—Continued.
Specific examples—Continued.
Thio-acetyl-benzyl
Mercapto-phenyl
Mercapto-benzyl
Mercapto-ethyl
Mercapto-butyl
Mercapto-benzoyl
Mercapto-stearoyl
Mercapto-thio-benzoyl
Mercapto-thio-stearoyl
Mercapto-thio-stearoyl-phenyl
Mercapto-thio-benzoyl-phenyl
Lauryl mercapto-phenyl
Lauryl mercapto-benzyl
Thienyl
Thiazyl

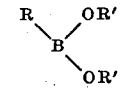
It is to be understood that the foregoing organic radicles may have one or more atoms of halogen, and more specifically of chlorine, substituted for one or more hydrogen atoms in the preparation of the halogen bearing esters which comprise the addition agents of this invention.

Esters of the type RR′BOR″, or R_2BOR'' may be made by replacing one ester group of a triester by reacting the ester with only enough Grignard reagent of the formula RMgBr to carry the reaction just one step. The second step may then be carried out with the same Grignard reagent, or a different one, R′MgBr, depending on whether R and R′ are to be the same or different.

The esters of type $RB(OR')_2$ may be prepared as follows:



The triester is reacted with RMgBr→



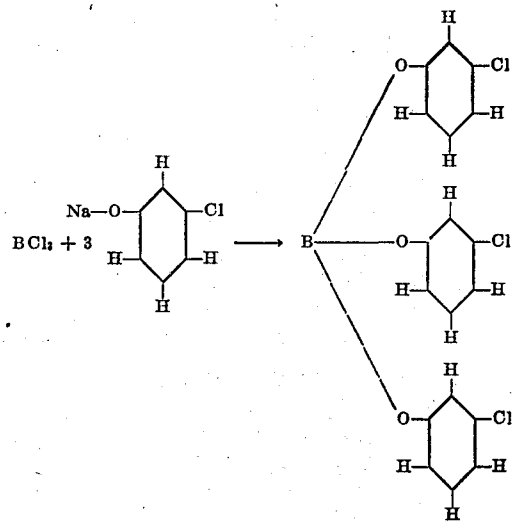
R and R′ may be any organic radicles, for example ethyl and $CH_2CH_2CH_2CH_2$—Cl respectively.

The above type reaction makes use of the Grignard reagent and reaction. The above reaction would give a product in which the R's are the same.

It is possible to use phenyl, chlorphenyl, naphyl, aliphatic, aliphatic substituted aromatic, and aromatic substituted aliphatic radicles to form the triester. It is preferable to have the chlorine in the radicles prior to the reaction.

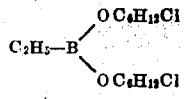
By varying the alcohol and the Grignard reagent it would be possible to obtain a great variety of esters.

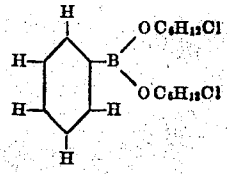
An aromatic radicle may be placed in the ester linkage in the following way:

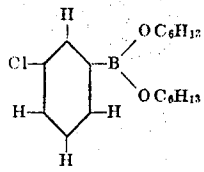


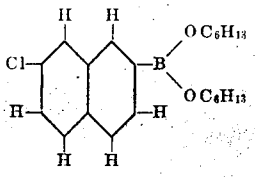
This triester can be reacted with any Grignard reagent to obtain the ester containing a carbon to boron linkage.

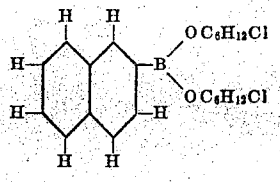
Specific examples of the organo-boron esters of this invention are:

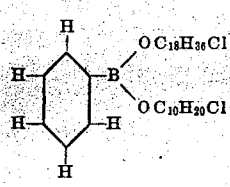


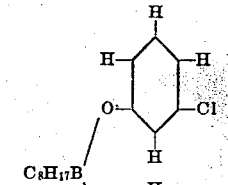


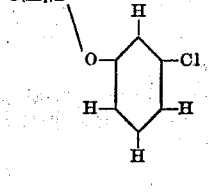


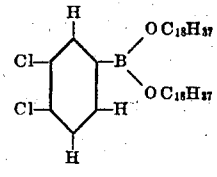


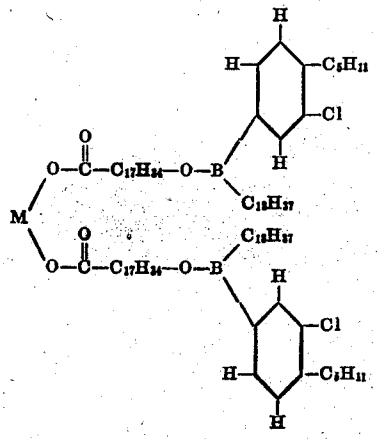












where M is a divalent metal, e. g., the alkaline-earth metals.

It is sometimes advantageous to combine more than one of these compounds in a blend to obtain particular properties. We accomplish this by mixing two or more of these compounds together and blending the mixture with hydrocarbon oil, or by blending one in the hydrocarbon oil, blending the second into this mixture, and so on until the composition is complete.

We have found that the organic boron compounds above set forth may be more effective in some compositions if a comparatively small amount of halogen-free, oxygen-containing carbon compound is included in the composition. The oxygen-containing carbon compound may be of aliphatic, aromatic, or hetero-cyclic nature and may be blended with the organic boron compound in amounts ranging from 5 to 50 per cent.

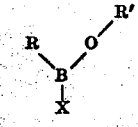
It will be understood that certain features and sub-combinations are of utility and may be employed without reference to other features and sub-combinations. This is contemplated by and is within the scope of our claims. It is further obvious that various changes may be made in details within the scope of our claims without departing from the spirit of our invention. It is, therefore, to be understood that our invention is not to be limited to the specific details shown and described.

Other modes of applying the principle of the invention may be employed, change being made as regards the details described, provided the features stated in any of the following claims or the equivalent of such be employed.

We therefore particularly point out and distinctly claim as our invention:

1. A lubricant comprising in combination a major proportion of a hydrocarbon oil and from .001% to about 20% of an oil-soluble halogen-bearing ester containing at least one carbon-boron bond.

2. A lubricant comprising in combination a major proportion of a hydrocarbon oil and from .001% to about 20% of an oil-soluble halogen-bearing organo-boron ester containing at least one carbon-boron bond and having the formula:



in which R and R' are organic radicles, X is a radicle of the class consisting of —R'' and —O—R''' where R'' and R''' are organic radicles.

3. A lubricant comprising in combination a

major proportion of a hydrocarbon oil and from .001% to about 20% of an oil-soluble halogen-bearing organo-boron ester containing at least one carbon-boron bond and having the formula:

$$R^d{=}B{-}OR$$

in which R^d is a divalent organic radicle and R is an organic radicle.

4. A lubricant comprising in combination a major proportion of a hydrocarbon oil and from .001% to about 20% of a halogen-bearing organo-boron ester containing at least one carbon-boron bond and having the formula $R_nB(OR')_{3-n}$ in which R and R' represent organic radicles and n is an integer less than 3.

5. A lubricant comprising in combination a major proportion of a hydrocarbon oil and from .001% to about 20% of a halogen-bearing organo-boron ester containing at least one carbon-boron bond and having the formula $RB(OR')_2$ in which R and R' are organic radicles.

6. A lubricant comprising in combination a major proportion of a hydrocarbon oil and from .001% to about 20% of a halogen-bearing organo-boron ester containing at least one carbon-boron bond and having the formula $R_2B.OR'$ in which R and R' are organic radicles.

7. A lubricant comprising in combination a major proportion of a hydrocarbon oil and a minor proportion of an oil-soluble halogen-bearing organo-boron ester containing at least one carbon-boron bond and containing an alkaline-earth metal in the molecule.

8. A lubricant comprising in combination a major proportion of lubricating oil and from .001% to about 20% of chlorphenylboric acid dibutyl ester containing at least one carbon-boron bond.

9. An extreme pressure lubricating composition comprising a major proportion of a lubricating oil and from .75% to about 20% of a halogen-bearing organo-boron ester containing at least one carbon-boron bond.

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