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LUBRICATING COMPOSITION

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This invention relates to improved lubricants, such as oils, greases or semi-fluid oils, containing as a major lubricating component hydrocarbon oil of natural or synthetic origin. In particular, the invention relates to lubricating compositions of this type having improved extreme pressure characteristics.

Requirements upon lubricants by developments in mechanical design are rigid and drastic. In many instances pressures at bearing surfaces are higher than those which petroleum oils will normally withstand. Illustrative are the conditions encountered in the lubrication of automotive hypoid gears, heavily loaded mill pinion gears, naval turret gears and the like. In many machines operation is, at least in part, under conditions of thin film lubrication or under similar conditions where film strength, oiliness and anti-wear characteristics of high order are required of the lubricating medium.

The principal object of this invention is the provision of a new composition of matter consisting of a compounded lubricating product possessing extreme pressure characteristics of a high order. Further objects include the provision of a compounded lubricant containing as a major hydrocarbon lubricating component which may be oil or grease of natural or synthetic origin, the lubricant having a superior combination of high film strength, oiliness and anti-wear characteristics.

In accordance with this invention these objects are achieved in substantial part by providing a lubricant, the essential lubricating components of which are a major amount of a hydrocarbon lubricating component which may be oil or grease and a minor amount of a body of boron fluoride and a substance selected from the class consisting of aliphatic, aromatic and heterocyclic mono- and polyamines, amino alcohols and amides more specifically described below. The proportion of this body to the hydrocarbon component should be such, however, that the mixture contains at least 0.001% by weight of boron fluoride (BF_3) while, on the other hand, more than 3% does not improve the lubricant. Within this range it has been found that the best results are obtained by providing from 0.1 to 1.0 percent of boron fluoride in the final lubricant mixture. The choice of the particular body to be employed and its boron fluoride content will of course depend on such matters as cost, ease of mixing and the nature of the lubrication problem.

The principal advantages and effectiveness of this new compounded lubricant are maintained

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when, in addition to the above mentioned essential components there are likewise present in the lubricating composition other components added to improve some specific characteristic or property thereof, such as viscosity, pour point, oiliness, flow, anti-wear characteristics, film strength or the like, the effect of the defined boron fluoride-containing body, when similar, being largely additive. Such diluents or additions may therefore, for the purposes of this invention be regarded as a non-essential portion of the composition, regardless of their specific beneficial or functional effect. The same may be said of substances known as extreme pressure additives, such as substances composed of chlorine, sulphur or lead containing compounds or materials, it being observable that the presence of the defined boron fluoride-containing bodies is effective to increase the extreme pressure characteristics of the compositions even when other extreme pressure additives are present.

The organic BF_3 body, which in accordance with this invention is blended with the hydrocarbon component, may be prepared in various ways but is, for the most part, conveniently prepared by exposing the amine, amino alcohol or amide to direct contact with gaseous BF_3. This can be easily accomplished under ordinary atmospheric pressure by bubbling the gaseous BF_3 through the amine, amino alcohol or amide in a liquid state. If the organic material is not liquid at room temperature it should be heated to and maintained at a temperature slightly above its melting point, say 5 or 10° C. Although the rate of flow of the gas is not critical with respect to formation of the BF_3 additive yet the flow should be controlled to avoid undue loss. The rate of acceptance of the BF_3 by the amine, amino alcohol or amide is improved if the liquid is stirred as the gas is introduced. The amount of BF_3 accepted by the amine, amino alcohol or amide is indicated by the increase in weight of the treated mass. The introduction of the gas may therefore be continued until a predetermined increase in weight, short of saturation, has been obtained or the flow of gas may be prolonged until no further increase in weight is observed at which point the amine, amino alcohol or amide is considered to be saturated with BF_3. Generally, it is most convenient to follow the latter procedure. In both cases, however, the length of time required to reach the end point can be easily determined for the material being treated and thus make it possible to introduce the desired amount of BF_3 without having to test a charge at intervals during the period of treatment. Although each amine, amino alcohol or amide may have a different saturation point or the desired BF_3 content may differ for various materials, this does not detract from the value of the BF_3-containing body in improving the load bearing quality of a hydrocarbon lubricant.

Other methods of introducing BF_3 can be employed, of course. For example, the amine, amino alcohol or amide may be exposed to the gas at higher temperatures and pressures and thus shorten the time required to introduce the desired amount of BF_3. Naturally, the temperature and pressure should be such that the amine, amino alcohol or amide is not decomposed to any substantial degree. Another method of contacting the organic material with BF_3 is to add compounds to it which can be decomposed with the release of BF_3. Such compounds as NH_4BF_4, $BF_3 \cdot NH_3$, the heavy metal fluoborates or even other organic materials previously treated with BF_3 may be employed. To effect decomposition it is usually necessary to heat the mixture of amine, amino alcohol or amide and the BF_3-containing substance. Further, it may be desirable to remove any residues resulting from the decomposition.

The amount of BF_3 that can be introduced into different amines, amino alcohols or amides will vary, as mentioned above, however, from 10 to 20 percent by weight is preferred where the saturation point permits it. In any case, the amines, amino alcohols or amides should be treated with sufficient fluoride to provide the desired BF_3 content in the lubricating composition as only a minor proportion of the additive is employed.

The resultant product may, in some instances, be an actual compound, there being some evidence to indicate that such is its nature, or it may be an additive or coordinate compound or even a solution or dispersion. I prefer, however, to term the product a body or composition consisting essentially of a substance selected from the class consisting of aliphatic, aromatic and heterocyclic mono- and polyamines, amino alcohols and amides and BF_3 and, therefore, not to characterize the state of association of the fluoride and the organic substance. In any event, the aliphatic, aromatic and heterocyclic mono- and polyamines, amino alcohols and amides act as acceptors of BF_3 to produce a body or substance having positive advantages as an additive in lubricating compositions.

The following list of amines, amino alcohols and amides is illustrative of the class which when treated with BF_3 produce the bodies just described: monoamines such as those of propyl through octadecyl and diethylhexylamine; symmetrical and unsymmetrical (mixed) alkyl polyamines such as propyl through octadecyl di- and triamines, 1,4 diaminobutane, 1,12 diaminododecane, ethylene diamine, diethylenetriamine, triethylenetetramine, tetraethylenepentamine and propylenediamine; aminoethanols such as monoethanolamine, diethanolamine, triethanolamine, dimethylethanolamine, diethylethanolamine, aminoethylethanolamine, methyldiethanolamine, phenylethanolamine, phenyldiethanolamine and ethylphenylethanolamine, also triisopropanolamine, ethyl phenyl diisopropanolamine, condensation products of high molecular weight amines with polyglycol derivatives of ethylene oxide and its homologues; amides and imides such as acetamide through octadecylamide, amides of the dicarboxcyclic acids, oxamide, succinamide, octadecylacetamide, n-propyloleamide, methylolstearamide, methylenedistearamide, benzylacetamide, methyl-x-naphthylamide, adipamide, succinimide, stearanilide, benzamidine, acetamidine, phenetidide, tolinidide, hydroxyacetanilide, acetocetanilide, chloracetocetanilide, dichloracetoacetanilide and acetoacet-o-toluidide; aromatic amines such as phenyltridecylamine, benzymethylamine, aminodiphenylamine, phenylheptadecylamine, and acetyl p-phenylenidiamine; heterocyclic amines such as pyridine and oxazoles, the derivatives and substituted derivatives of the oxazoles such as dihydrooxazole and its alkyl substituted derivatives, for example 2-tridecyl-4-(hydroxymethyl)-4-ethyl oxazoline and 2 hendecyl oxazoline; as well as other amines such as 2-hydroxyethylamine, heptadecamethylenimine, hexamethylenimine, 16-hydroxyhexadecylamine, octadecadienylamine, phenyl biguanide, pyrinidine, 2-substituted-4-amino-6-N phenyl-amino-1,3,5-triazine, 12-(dibutylamino) dodecananidine, lauranidoxine, hydroxamic acids of propionic, lauric and stearic acids, phenylhydrazides of lauric and stearic acids, piperidides of acids, aminonaphthol, aminophenylglycine, aminoquinoline, aniline, anisidine, arabinose phenylhydrazone, benzoylhydrazine, benzoylthiourea, benzylhydroxylamine, carbohydrazide, acetaldazine, acetaldoxime, acetaldehyde phenylhydrazone, acetanilide, acetophenone phenylhydrazone, acetyl phenylhydrazine, aminopropionic acid, aminoacetophenone, aminoanthraquinone, aminoazobenzene, aminodibutylaniline, aminoethylacetamilide, urea, quinoline, morpholine, tetraethanolammonium hydroxide and phenylmethylpyrazolone.

The introduction of the BF_3 into the amine, amino alcohol or amide may change the color thereof, often making it darker. Also, the liquid amine, amino alcohol or amide may become thicker as the introduction of BF_3 proceeds and the melting point of the end product may be higher than that of the original amine, amino alcohol or amide. Such changes do not affect the advantages gained from the presence of the BF_3-containing bodies in a hydrocarbon lubricant, certainly the results of the invention do not depend upon or are responsive to any peculiar chemical or physical qualities of the BF_3-containing additive, rather they depend upon the admixture of the additive with the hydrocarbon lubricant.

The action of these organic BF_3 bodies in a hydrocarbon lubricant is positive and specific to increase the load bearing capacity of the lubricant and, in many instances, to definitely improve its anti-wear characteristics. The amount of these organic BF_3 bodies necessarily present to produce these specific effects is very small. The amounts used, should in any case, as mentioned above, provide at least 0.001% by weight of BF_3 in the final mixture. The amount of any given BF_3-containing body desirably present to produce optimum effects in any particular hydrocarbon lubricant can be readily determined by simple trial, the optimum usually being found within the range of 0.5 to 5 percent by weight of the amine, amino alcohol or amide BF_3 body. Amounts below about 0.01 percent by weight often do not produce sufficient effect to be usually characterized as commercial, but the effect produced is such as to be useful under exceptional conditions. One may use a relatively small amount of an additive containing a substantial quantity of BF_3 or a larger amount of the same or another additive which contains a smaller proportion of BF_3 to introduce a given quantity of BF_3 into the hydrocarbon component. In any event, the organic BF_3 additive forms a minor amount or proportion of the lubricating composition.

The load bearing qualities of a lubricant may be relatively determined by various methods. The method used to obtain the comparative data hereinafter set forth is practiced on a Falex machine and consists in submitting small quantities of the lubricant to the action of a rotating steel journal rotated in contact with two V-shaped bearing blocks. During testing measurable pressure was applied to the rotating journal by use of an automatic loading device. The two bearing blocks and the journal were submerged in the lubricant sample throughout the tests. In each test load was applied and allowed to reach 500 pounds; at this load the journal was then run for 5 minutes; thereafter the load was gradually increased until lubrication failure occurred. The load at the time of failure is designated as the failure load. In general, the failure loads are reproducible within 150 pounds at lower pressure and 300 pounds at higher pressure; the results are not absolute but are comparative with a standard run under the same conditions. In the tests by which the results set forth in the following table were secured, the hydrocarbon oil was in all cases the same, being a straight mineral oil. In all cases 1 percent by weight of additive was present in the oil. The additive had been prepared by passing BF_3 through the amine, amino alcohol or amide until the weight of the body became substantially constant, that is, the organic material became saturated with the BF_3, as has been described hereinabove. Each comparative test was run on two samples, one sample being the oil containing 1 percent by weight of an amine, amino alcohol or amide, the other sample being the oil containing the same weight of a BF_3-containing body. In the first column of the table the nature of the additive is set forth, in the second column is listed the BF_3 content of the treated material, in the third column is the BF_3 content of the lubricant, in the fourth column the failure load in pounds is given and in the fifth column is shown the percentage increase in failure load caused by the presence of the BF_3-containing body.

Additive	Percent BF_3 in Additive	Percent BF_3 in Lubricant	Failure Load in Pounds	Percentage Increase
Acetamide	----	----	900	[1] 344
Acetamide + BF_3	42.7	.43	[1] 4,000	
Stearamide	----	----	650	269
Stearamide + BF_3	18.	.18	2,400	
Tetraethylene pentamine	----	----	1,050	13
Tetraethylene pentamine + BF_3	43.	.43	1,190	
Monoisopropanolamine	----	----	850	35
Monoisopropanolamine + BF_3	52.	.52	1,150	
2 Amino, 2 methyl, 1,3 propanediol	----	----	850	29
2 Amino, 2 methyl, 1,3 propanediol + BF_3	30.	.3	1,100	
Triisopropanolamine	----	----	850	229
Triisopropanolamine + BF_3	24.	.24	2,800	
Urea	----	----	950	178
Urea + BF_3	53.	.53	2,650	
Quinoline	----	----	1,050	116
Quinoline + BF_3	34.	.34	2,270	
Morpholine	----	----	650	95
Morpholine + BF_3	33.	.33	1,270	

[1] Greater than.

The machine on which the tests were run had a capacity of 4500 pounds. Where this capacity was approximated or exceeded the failure load is indicated above as being greater than 4000 pounds. These tabulated results are indicative of the improvement obtained by the practice of this

invention. In general, torque and temperature values obtained during these tests indicated that the organic BF_3 body improved friction qualities and observations simultaneously made indicated increases in anti-wear characteristics.

In compounding lubricant compositions of the type herein described and claimed the organic BF_3 additive may merely be mixed with the hydrocarbon lubricants, the properties of which are to be improved. Where the additive is insoluble in the lubricant or it is desired to incorporate therein amounts in excess of solubility, emulsions or dispersions may be prepared according to known principles.

This application is a continuation-in-part of my prior application Serial No. 714,911 filed December 7, 1946, and entitled Lubrication Composition, now abandoned.

Having thus described my invention and the known advantages thereof, I claim:

1. A lubricating composition comprising a major proportion of a hydrocarbon lubricating oil blended with a minor proportion of a body consisting essentially of a substance selected from the class consisting of aliphatic, aromatic and heterocyclic mono- and polyamines, amino alcohols and amides and BF_3, said body being prepared by exposing said substances to direct contact with a substance selected from the class consisting of gaseous BF_3 and decomposable compounds yielding gaseous BF_3 and being present in such amount that the lubricating composition contains from 0.001 to 3 per cent by weight of BF_3, said lubricating composition possessing extreme pressure characteristics superior to those exhibited by a similar composition in which the minor proportion aforesaid consists of said substances selected from the class consisting of amines, amino alcohols and amides.

2. A lubricating composition comprising a major proportion of a hydrocarbon lubricating oil blended with a minor proportion of a body consisting essentially of an oxazole and BF_3, said body being prepared by exposing said oxazole to direct contact with a substance selected from the class consisting of gaseous BF_3 and decomposable compounds yielding gaseous BF_3 and being present in such amount that the lubricating composition contains from 0.001 to 3 per cent by weight of BF_3, said lubricating composition possessing extreme pressure characteristics superior to those exhibited by a similar composition in which the minor proportion aforesaid consists of an oxazole.

3. A lubricating composition comprising a major proportion of a hydrocarbon lubricating oil blended with a minor proportion of a body

consisting essentially of an oxazoline and BF_3, said body being prepared by exposing said oxazoline to direct contact with a substance selected from the class consisting of gaseous BF_3 and decomposable compounds yielding gaseous BF_3 and being present in such amount that the lubricating composition possessing extreme pressure characteristics superior to those exhibited by a similar composition in which the minor proportion aforesaid consists of an oxazoline.

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