Bergeron

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[54]	GREASE	COMPOSITION
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		232/39, 13
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[57] ABSTRACT

Disclosed herein are lubricating compounds comprising the reaction product of significant amounts of a fish oil, a sulfur-modified sperm oil, a microcrystalline wax and an oxide of lead, preferably litharge. In one embodiment, the lubricating compounds additionally comprise up to 90% by weight of a hydrocarbon oil which results in an extreme pressure grease lubricant having desirable lubricating properties. Also disclosed herein is a process for preparing the lubricating compounds of the invention.

26 Claims, No Drawings

GREASE COMPOSITION

BACKGROUND OF THE INVENTION

The invention relates to lubricating compounds and uses therefore. In one embodiment the invention re- 5 lates to extreme pressure lubricants employed to reduce wear on moving parts subjected to frictional wear under high bearing pressures.

faces in, for example, mechanical bearings, anti-To reduce friction and protect the contacting surfriction bearings and journals, lubricants such as viscous oils and greases are applied to the contacting surfaces. These lubricants must themselves withstand the extreme pressure exerted on them by the contacting surfaces without substantially losing their lubricating quality. Where extreme pressure greases are involved, it is necessary to provide a gelling agent or thickener which thickens a hydrocarbon oil and an extreme presrous and chlorine and lead soaps which allows the grease to stand up under lubricating conditions. An improved form of such a grease composition is disclosed in U.S. Pat. No. 3,652,415. In the lubricating compound of the patent, a grease is prepared by thickening 25 a hydrocarbon oil with a metallic stearate soap and adding thereto an extreme pressure proportion of a fish oil lead soap and a sulfur-modified sperm oil. It is also known in the art to add other extreme pressure agents to lubricating compounds. Although the lubricating 30 compound of the patent exhibits desirable extreme pressure lubricating properties, it employs a gelling. agent as well as an extreme pressure agent in order to achieve the desired lubricating composition.

It is desirable to provide a lubricating compound for 35 use in an extreme pressure environment which does not require the addition of discrete gelling agents and extreme pressure additives to the same composition. This is desirable not only from the standpoint of economy but also from the fact that many extreme pressure additives interreact with gellation agents so that these agents mutually interfere with their respective functions.

In many lubricating applications, such as extreme 45 pressure greases and open gear lubricants, it is desirable to have lubricating compounds which are nonnewtonian in nature. Non-newtonian lubricating compounds exhibit a broad apparent viscosity range and this can be used in a broad range of applications which 50 are not suitable to newtonian fluids.

Moreover, where greases or grease-like lubricants are involved it is also desirable to have a lubricating compound which is thixotropic in nature and has a reversible gel structure, i.e., will regel after melting. Re- 55 versible gel greases and grease-like substances are desirable where it is preferred to apply the lubricating compound as a liquid but for it to be in a gel or gel-like state under operating temperatures and conditions. Most greases, however, possess only a low degree of thixotropicy and have non-reversible gel structures. Therefore, they cannot be heated above their liquification or drop point temperature since they are incapable of returning to their gel structure upon cooling. Similarly when such greases are exposed to shear, their gel structure does not completely return when shear is removed.

SUMMARY OF THE INVENTION

In view of the foregoing it was desired to provide lubricating compounds having reversible gel structures, which are non-newtonian and thixotropic in nature.

The lubricating compounds of the invention comprise the reaction product of significant amounts of a fish oil, a sulfur-modified sperm oil, a microcrystalline wax and an oxide of lead. In some embodiments of the prise a hydrocarbon oil.

In one preferred embodiment the weight ratio of fish oil to the oxide of lead is from about 1:2 to about 2:1; the weight ratio of the lead soap, i.e., fish oil plus the 15 oxide of lead to sulfur-modified sperm oil is from about 1:1 to about 4.4:1; and the weight ratio of the microcrystalline wax to the lead soap is from about 1:2 to about 6.5:1.

Within the above ratios the amount of microcrystalsure additive such as compounds of sulfur, phospho- 20 line wax in the compound, on a total compound weight percent basis, should not exceed from about 80 to about 85 percent, at which point the wax begins to separate from the lubricating compound. Within the specified ratio of fish oil to oxide of lead, the oxide is present in an amount in excess of the amount which would be required to react with the fish oil to produce a normal lead soap. Preferably, the oxide of lead is present in the maximum amount that will chemically combine with the other components of the lubricating compound. This amount may readily be determined for any particular composition. Surprisingly, even though lead soaps are not good gelling agents, the above composition of fish oil lead soap, microcrystalline wax and sulfurmodified sperm oil results in a thixotropic grease-like lubricating compound having a reversible gel structure and which exhibits particularly desirable extreme pressure lubricating characteristics.

In another preferred embodiment of the invention, hydrocarbon oil may be added to the composition to form a lubricating grease having desirable apparent viscosity, thixotropic and extreme pressure characteristics. In this embodiment, the hydrocarbon oil is present in an amount of up to about 90 percent of the total weight of the resulting lubricating compound.

The lubricating compound of the invention may be prepared according to the following process of the invention. First, the fish oil, the sulfur-modified sperm oil and the microcrystalline wax are added to a reaction vessel. The mixture is heated to a reaction temperature of at least about 300°F. The oxide of lead is added to the heated mixture in the amount set out above. The temperature is maintained in the range from about 300°F to about 400°F for a sufficient time so that all of the lead oxide reacts with the other components of the mixture to form the lubricating compound product. The upper limit of the temperature of the reaction is determined by the point at which metallic lead begins to drop out of the solution and may vary depending on the properties of the particular mixture. The lower limit of temperature is that temperature at which the oxide of lead will react with the other components in a significant amount. The above compound may then be added, preferably with heating, to a hydrocarbon oil to 65 form a grease.

DETAILED DESCRIPTION OF THE INVENTION

In one embodiment of the invention the lubricating

compound comprises the reaction product of a fish oil, a sulfur-modified sperm oil, a microcrystalline wax and an oxide of lead. The fish oil may be any crude fish oil such as, for example, tuna oil, herring oil, menhaden oil, sardine oil, cod-liver oil, halibut oil, salmon oil, and 5 shark oil. The primary requirement is that the particular fish oil employed must be a semi-drying oil, i.e., containing both saturated and unsaturated oils. The sulfur-modified sperm oil may be either synthetic or rized sperm oil or sullfonated sperm oil. In one embodiment the fish oil is crude tuna oil, the sperm oil is Lubrizol No. 5003, sold by the Lubrizol Corporation of Cleveland, Ohio and the oxide of lead is litharge.

1:2 to about 2:1 and is preferably about 1.3:1 when litharge is used, the fish oil is crude tuna oil and an extreme pressure lubricant is desired. The ratio of fish oil to the oxide of lead will vary not only depending upon position but may well vary from batch to batch of the same type of fish oil. This result obtains since the quantity of the oxide of lead required for a particular fish oil is dependent both upon the fatty acid content of the fish oil, as measured by its neutralization number, and 25the polyunsaturate content, as measure by its iodine value. The ratio of lead soap to sulfur-modified sperm oil may be from about 1:1 to about 4.4:1 and is preferably about 2.3:1 when litharge is used, the fish oil is crude tuna oil and an extreme pressure lubricant is de- 30 sired. The ratio of microcrystalline wax to lead soap may be from about 1:2 to about 6.5:1 and is preferably about 1.3:1 when litharge is used, the fish oil is crude tuna oil and an extreme pressure lubricant is desired.

The lubricating compounds of the invention compris- 35 ing the reaction product of the above components within the specified ratios are homogenous, solid, greaselike substances having a gel structure, are thixotropic and exhibit non-newtonian flow characteristics. Particular features of the lubricating compounds of the invention are that they are extremely thixotropic in nature and that they will return to their original gel structure even after they have been heated above their drop point or have been subjected to shear. This is in direct contrast to most lubricants of this type, like conventional greases, which normally are much less thixotropic in nature and do not completely regain their gel structure after being subjected to shear. The nonnewtonian nature of the lubricating compounds of the invention results in a broad range apparent viscosity which makes them suitable for use over a broad range of applications for which newtonian fluids are not suited.

Within the ratios set forth above the reactants which form the lubricating compounds of the invention are selected so that the above described excess of litharge is present and that the maximum microcrystalline wax content does not exceed about 80 to about 85 percent by weight. Above that point the microcrystalline wax will not remain a part of the compound but separates out.

Although the exact mechanism by which the lubricating compounds of the invention obtain and exhibit the useful properties thereof is not known, it is believed that the nonnewtonian nature of the compositions and the fact that they are thixotropic is related to the reaction of the oxide of lead with the fish oil which allows

close association to the sulfur groups of the sulfurmodified sperm oil. This is believed to allow coupling between the microcrystalline wax and the fish oil lead soap. This reaction is believed to be related to the iodine number of polyunsaturate content of the fish oil in that a portion of the lead is believed to react with the fish oil at the unsaturated sites.

In order to obtain the lubricating compounds of the invention the amount of the oxide of lead added to the natural sulfur-modified sperm oil, as for example, sulfu- 10 composition is in excess of that required to produce a normal lead soap by direct reaction with the fish oil alone. By "normal lead soap" is meant the soap produced by the reaction of a fish oil with that amount of an oxide of lead which is necessary to neutralize the The fish oil to oxide of lead ratio may be from about 15 acid components of the fish oil. When the lead oxide is litharge, the amount in excess is preferably the maximum amount of litharge that will combine with the fish oil, sulfur-modified sperm oil and microcrystalline wax in the particular composition. As will be appreciated by the particular fish oil employed in the lubricating com- 20 the art skilled this amount may readily be determined experimentally for particular compositions. For example, once the quantities of the other reactants is determined a small sample batch may be prepared and the oxide of lead added thereto until no further oxide will react. Since the oxide is a dense solid which settles out of the mixture the unreacted portion may readily be recovered and weighed. The amount of the oxide which reacted may, therefore, be determined.

In yet another embodiment of the invention, a hydrocarbon oil may be added to the lubricating compound described in an amount such that the hydrocarbon oil comprises up to about 90 percent by weight of the resulting lubricating compound. The resulting composition is a lubricating grease possessing the properties described above. Among the hydrocarbon oils suitable for use in compositions of this embodiment of the invention there may be mentioned mineral oils such as residual oils, spindal oils, pale oils, neutral oils, bright stock, black oil and other lubricating oils known to those skilled in the art which may be untreated, conventionally refined, solvent treated by known processes and hydrofinished or hydrofined. Synthetic oils such as polybutenes, esters and other synthetic oils predominately composed of hydrocarbons known to the lubricating art also fall within the scope of the term hydrocarbon oil as defined herein. Particular oils that may be mentioned are MVI 400 Shell and MVI 150 Shell (bright stock).

The exact amount of hydrocarbon oil in the total lubricating compositions of this embodiment of the invention is dependent upon the gelling characteristics of the hydrocarbon and the end use desired. For example, where an open gear lubricant is desired, asphaltic residual oil in an amount of 49.8 percent of the mixture is added.

The lubricating compounds of the invention may be beneficially employed in a wide variety of conventional lubricating uses. An exceptional characteristic of the lubricating compounds of the invention is that they may be used in extreme pressure service as an extreme pressure lubricant without the further addition of any of the usual extreme pressure additives beyond what is necessary to form the gel structure. Ordinarily, to obtain greases useful in an extreme pressure environment it is necessary to add gelling agents as well as extreme pressure additives to the grease composition. However, most extreme pressure additives and gellation agents

known to the art, when present together in grease compositions, interfere with each other's functions. Thus, the effectiveness of both agents is reduced.

Commonly used gelling agents are the metallic stearate based soaps such as those disclosed in the U.S. Pat. 5 No. 3,652,415, the disclosure of which is incorporated by reference herein. In the composition of the patent a fish oil lead soap and sulfur-modified sperm oil extreme pressure agent is added to an oil thickened to grease consistency with a metallic stearate soap. How- 10 ever, in the lubricating compounds of the invention herein, it is not necessary to employ a metallic stearate soap or other gelling agent. Rather, surprisingly, it has been found that the lubricating compounds of the invention may function in extreme pressure environ- 15 ments as extreme pressure lubricants without the addition of any other gelling agents or extreme pressure additives.

The lubricating compounds of the invention may be prepared according to the process of the invention by 20 ing element bearings. the following steps. First, the fish oil, sulfur-modified sperm oil, and microcrystalline wax is added to a reaction vessel. The mixture is then heated, with mixing, up to the reaction temperature which will vary depending on the properties of the components. For example, when the fish oil is crude tuna oil, the mixture should be brought to a temperature of from about 300°F to about 400°F. The upper limit of the temperature is determined by the point at which the oxide of lead to be added in the next step of the reaction drops out of the 30 reaction product as metallic lead. The lower limit is determined by the point at which the oxide of lead will not react at a significant rate with the other components.

Next, an oxide of lead is added to the heated mixture 35 in an amount in excess of that which will form a normal fish oil lead soap as described above. Preferably, the oxide of lead is present in the maximum amount that will react with the mixture. The components are allowed to react to form the lubricating compound for a time sufficient to react all of the lead oxide present or until no further lead oxide will react. The reaction product is then cooled to form a solid grease-like substance. In one embodiment of the invention the greaselike substance may be melted into a hydrocarbon oil which may form up to 90 percent by weight of the resulting mixture. Alternatively, the hydrocarbon oil may be added to the hot mixture. In either case the mixture is cooled to form a lubricating grease.

The lubricating compounds of the invention may be employed in a wide variety of lubricating uses due to the many desirable properties exhibited by these lubricating compounds as described above. The reversible gel nature of the compounds allows them to be applied as a liquid which is desirable in many applications such as open gear lubrication. The lubricating compounds of the invention are particularly useful in extreme pressure applications in that they meet extreme pressure specifications without the addition of any additional extreme pressure additives or any other gellation or thickening agents. Thus, there is obtained in one composition a lubricating compound which possesses both highly desirable thixotropic and reversible gel properties, as well as extreme pressure properties. Particularly in the compounds of the invention containing hydrocarbon oil, an extreme pressure grease is obtained which does not require the addition of a metallic stea-

rate soap or any other thickener or any additional extreme pressure agents.

The lubricating compounds of the invention also exhibit excellent resistance to aqueous fluids such as would be encountered in operations exposed to salt water spray or steam cleaning. The thixotropic property of the lubricating compounds and the high apparent viscosity associated therewith prevents dust and dirt from becoming embedded in the lubricating compounds. The lubricating compounds also displace water on the surfaces to be lubricated thereby wetting the bearing surfaces with the lubricating compound.

More dilute versions of the lubricating compounds of the invention may be used as cable steel lubricants since the properties of the lubricating compounds allows them to be carried to the core of the cable. This property is especially useful where steel cables have a hemp core. The extreme pressure properties also make some compositions suitable for large slow moving roll-

It is within the scope of the invention to add to the lubricating compounds of the invention, additives well known to the art to obtain particular properties desirable for some applications. For example, molybdenum disulfide, tungsten disulfide, zinc ozide and graphite may be added to the lubricating compound to impart low starting torque properties to the lubricating compound.

The lubricating compounds and processes of the invention are further described in the following example wherein all parts are by weight and all degrees in degrees fahrenheit unless otherwise specified. In the examples the symbols used to express ratios of the reactants have the following meanings:

Fo identifies fish oil;

SSO identifies sulfur-modified sperm oil;

L identifies litharge

MCW identifies microcrystalline wax; and

FOLS identifies fish oil lead soap which is the weight total of fish oil and litharge in the composition.

Unless otherwise specified, the fish oil in the following examples is crude tuna oil wherein the carbon chains are normally C_{14} to C_{24} and which is a product of Van Camp Cannery, Los Angeles, California; the sulfur-modified sperm oil in Lubrizol 5003; the litharge is commercial grade; the microcrystalline wax is a mixture of commercial waxes having a 120° melting point; and the residual oil is No. 1 residual oil produced by the Newhall refinery at Newhall, California, having a viscosity of about 1050 SSU at 150°F. Residual oil was chosen since it is one of the most difficult hydrocarbon oils to gel.

EXAMPLE I

A lubricating compound having the following composition was prepared.

	COMPONENT	WEIGHT (OZ.)	RATIOS	
60	Fo MCW SSO L	13.0 30.9 9.9 10.0	Fo/L MCW/FOLS FOLS/SSO	1.3:1 1.34:1 2.3:1

The crude tuna oil, microcrystalline wax and sulfurized sperm oil were placed in a reaction vessel and heated to about 300°F. At the point the litharge was 20

sprinkled in the mixture and the mixture was maintained between about 310°F to about 340°F for about 1 hour while the reaction proceeded. The mixture was then cooled. The resulting lubricating compound was a hard, waxy, grease-like solid, which when exposed to 5 shear stress remained a grease-like semi-solid. This is a preferred compound of the invention and was used as a standard of comparison in the following examples.

The above lubricating compound was then melted into an equal weight of residual oil which resulted in a 10 gelled grease. The grease composition possessed excellent extreme pressure and thixotropic properties which render it suitable for use in applications requiring a NLGI No. 6 classified lubricating grease and particularly an extreme pressure lubricating grease. This composition represents a preferred embodiment of the hydrocarbon oil containing lubricating compound of the invention.

EXAMPLE II

A mixture was prepared from the following components.

COMPONENT	WEIGHT (OZ.)	RATIOS	
Fo MCW SSO L	15.1 36.0 1.4 11.5	Fo/L MCW/FOLS FOLS/SSO	1.3:1 1.4:1 19:1

The crude tuna oil, microcrystalline wax and sulfurized sperm oil was introduced into a reaction vessel and heated with mixing until the mixture reached about 300°. At this point litharge was sprinkled into the mixture and the mixture was maintained at between 310° and 340° for about 1 hour as the reaction proceeded. The mixture was then cooled. The resulting cooled composition was not homogenous in nature. Rather, wax was observed to separate from the rest of the mixture. The composition exhibited unacceptable lubricating compound properties. In order to obtain a lubricating compound with more acceptable lubricating properties, the above composition was mixed with an equal weight of the non-residual oil containing lubricating composition of EXAMPLE I giving a mixture with the following proportions and ratios:

COMPONENT	WEIGHT (OZ.)	RATIOS	
Fo MCW SSO L	28.1 66.9 11.3 21.5	Fo/L MCW/FOLS FOLS/SSO	1.3:1 1.3:1 4.4:1

The resulting mixture exhibited lubricating properties in the just acceptable range. A part of that mixture was melted into an equal part of residual oil which produced a gelled grease. The gel structure was mealy in nature and the shear stability of the grease was low as measured on a Portivis viscosimeter. The mixture had an initial viscosity of more than 4000 poise (the measuring equipment could not measure above 4000 poise). After five seconds this viscosity was reduced to less than 700 poise.

The foregoing example demonstrates the approximate upper limit of the ratio of lead soap to sulfurized sperm oil in a composition employing crude tuna oil as the fish oil of the lead soap. As will be readily appreciated by the art skilled, other maximum ratios may be established using other fish oils. It is within the scope of the invention to include other ratios of lead soap to sulfurized sperm oil where different fish oils are employed.

EXAMPLE III

A lubricating compound having the following components was prepared.

5	COMPONENT	WEIGHT	RATIO	\$
	Fo	11	Fo/L	1.4:1
	MCW	30	MCW/FOLS	1.6:1
	SSO	19	FOLS/SSO	1:1
	L	8		

The crude tuna oil, microcrystalline wax and sulfurized sperm oil were placed in a reaction vessel and heated with stirring to about 300°. Litharge was then sprinkled into the mixture which was maintained between 310° and 340° for about 1 hour until the reaction had gone to completion. The mixture was then cooled to ambient temperature. The resulting lubricating compound was a hard waxy solid which, when subjected to shear, quickly became a tacky liquid. The lubricating compound was melted into an equal weight of residual oil. The resulting compound exhibited a weak gel structure which upon shear gave a tacky liquid.

The foregoing example demonstrates the approximate minimum lead soap to sulfurized sperm oil ratio which will provide acceptable lubricating compounds when the fish oil is tuna oil. As will readily be appreciated by the art skilled this minimum ratio may vary with other fish oil. It is within the scope of the invention to include such variable ratios when other fish oils are employed.

EXAMPLE IV

A lubricating compound having the following composition was prepared.

COMPONENT	WEIGHT	RATIOS	
Fo	21	Fo/L	1.3:1
MCW	18.5	MCW/FOLS	1:2
SSO	18.5	FOLS/SSO	2:1
L	. 16	1-	

The crude tuna oil, microcrystalline wax and sperm oil were introduced into a reaction vessel and heated to about 300°. Litharge was then sprinkled into the mixture which was maintained with stirring between 310° and 340° for about 1 hour. The mixture was then cooled. The resulting lubricating compound was a grease-like substance having the general consistency of conventional grease. This compound was melted into an equal weight of residual oil resulting in a semi-liquid compound which indicates some lack of gel strength.

EXAMPLE V

A lubricating compound having the following composition was prepared.

COMPONENT	WEIGHT	RATIOS			
Fo	5	Fo/L	17:1		
MČW	52	MCW/FOLS	6.5:1		
SSO	4	FOLS/SSO	2:1		
L	. 3				

The crude tuna oil, microcrystalline wax and sulfurized sperm oil were added to a reaction vessel and heated to about 300°. At this point litharge was sprinkled into the mixture with stirring and the mixture maintained between 310° and 340° for about 1 hour. The mixture was then cooled. The resulting lubricating compound was a waxy solid. When the compound is melted into an equal weight of residual oil a lubricating compound having a relatively weak gel structure is produced which quickly returns to the liquid state under shear.

The foregoing example demonstrates the approxi- 20 mate maximum ratio of microcrystalline wax to lead soap which will still give an acceptable lubricating compound when the fish oil is crude tuna oil. As will readily be appreciated by the art skilled the use of alternative fish oils within the scope of the invention may well re- 25 sult in a different maximum ratio. It is within the scope of the invention to include compositions having a microcrystalline wax to lead soap ratio outside the above ratio when other fish oils are employed.

EXAMPLE VI

A lubricating grease was prepared according to the process of the invention having the following composi-

COMPONENT	WEIGHT (No.)	RATIOS	
Fo MCW SSO L Asphaltic Residual Oil	76.5 180.0 58.5 57.6 369.0	Fo/L MCW/FOLS FOLS/SSO	1.3:1 1.3:1 2.3:1

graphite were added to the lubricating grease. The asphaltic residual oil is 10 percent asphalt added to 90 percent residual oil. This grease represents a preferred embodiment of the invention possessing all of the desirable characteristics discussed above. It is particularly 50 useful as an open gear lubricant.

A lubricating grease of the invention was tested for wear characteristics by the LFW/1 standard load friction and wear test for extreme pressure greases. The grease was prepared in the proportions set forth in EX- 55 AMPLE VI but since asphaltic residual oils sometime have natural extreme pressure properties, MVI 150 bright stock was substituted for residual oil in the grease. MVI 150 was also used in the other comparison lubricants. The test was conducted with a base load of 60 180 pounds and at 60,000 pounds per square inch. The block material had a hardness of 34 Rockwell C and a surface finish of 15 RMS while the ring material had a hardness of 60 Rockwell C and a surface finish of 7 RMS. The test was conducted at 85 rpm for one hour. 65

The test results demonstrated that the composition of the invention compared favorably with other extreme pressure lubricating greases which contain additional

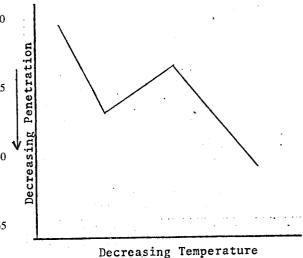
gelling agents or additives not present in the composition of the invention. In particular the wear characteristics are significantly better than those demonstrated by an extreme pressure grease prepared according to the disclosure of the above reference U.S. Pat. No.

The temperature and coefficient of friction curves of the lubricating compund during the test also demonstrated desirable properties of the lubricating compound. Thus, the temperature increased in the early part of the test and then returned to a lower value and remained steady for the rest of the test. Similarly the coefficient of friction decreased as the test continued until a sharp downward break was obtained in the curve at from about 2500 to about 3000 revolutions after which the coefficient of friction leveled off at a relatively low value below 0.02. Similarly the indicated wear demonstrated a leveling off at about 4000 revolutions at a level of about 0.001 inches.

The lubricating compound of the invention also compares favorably with a lubricant formulated with the percentage of additive normally used to pass the Gardner-Denver test for extra pressure lubricants. That test utilizes a pin-type Weeks oil tester and requires that the lubricant pass a 300,000 pound per square inch minimum before rapid wear or pin seizure occurs. However, the particular sample was not tested on the machine.

It will be noted that all of the desirable characteristics of extreme pressure lubricating greases are obtained 30 with the composition of the invention without the addition of any extra gelling agent or extreme pressure additive to the lubricating composition as is required in known extreme pressure lubricant compositions.

Lubricant compositions prepared according to the 35 invention were also subjected to standard ASTM penetration tests modified by performing the tests at various temperatures. These tests demonstrated that lubricating compositions of the invention possess penetration/temperature characteristics markedly different from those of conventional greases. That is, with conventional greases, the penetration/temperature relationship is substantially linear or a simple curve depending on graph plotting techniques. (Viscosity is normally plotted logarithmically to yield linear results). The lu-In addition 23.4 pounds of MoS₂ and 135 pounds of bricating compositions of the invention, on the other hand, exhibit a penetration/temperature relationship which may be generalized as follows:



As will be readily appreciated, this characteristic allows lubricating compositions of the invention within a particular NLGI grade to be used over a much broader temperature range in many applications than comparable lubricants of conventional formulations. In open gear lubrication this characteristic is particularly desirable.

The lubricants of the invention may be readily mixed with volatile solvents such as, for example, hydrocarbon solvents, chlorinated hydrocarbon solvents, alcohols, etc. Preferably the solvent may comprise up to about 50 percent by weight of the resulting mixture. As will be appreciated by the art skilled, such diluted mixtures may be combined with suitable propellants such as air, fluorinated ethylenes and other propellants known to the areosol art. The resulting areosol spray lubricant is particularly suitable for open gear lubrication applications as well as other applications where it is desirable to apply a lubricant as one would a paint. 20

- 1. A thixotropic lubricating compound having a reversible gel structure comprising the reaction product of significant amounts of a crude fish oil, a sulfurmodified sperm oil, a microcrystalline wax and an oxide of 25
- 2. The lubricating compound of claim 1 further comprising up to about 90 percent by weight of the total compound of a hydrocarbon oil.
- 3. The lubricating compound of claim 1 wherein the 30 crude fish oil is selected from the group consisting of crude tuna oil, herring oil, menhaden oil, sardine oil, cod liver oil, halibut oil, salmon oil and shark oil and the sulfur-modified sperm oil is selected from the group consisting of sulfurized sperm oil and sulfonated sperm 35 oil
- 4. The lubricating compound of claim 3 wherein, the weight ratio of the fish oil to the oxide of lead is from about 1:2 to about 2:1; the weight ratio of the fish oil plus the oxide of lead to the sulfur-modified sperm oil $\,40\,$ is from about 1:1 to about 4.4:1; and the weight ratio of the microcrystalline wax to the fish oil plus the oxide of lead is from about 1:2 to about 6.5:1.
- 5. The lubricating compound of claim 4 wherein the oxide of lead is litharge.
- 6. The lubricating compound of claim 4 further comprising up to about 90 percent by weight of the total compound of a hydrocarbon oil.
- 7. The lubricating compound of claim 4 wherein, when the fish oil is crude tuna oil, the ratio of the fish oil to the oxide of lead is about 1.3:1; the weight ratio the fish oil plus the oxide of lead to the sulfur-modified sperm oil is about 2.3:1; and the weight ratio of the mi-1.3:1.
- 8. The lubricating compound of claim 7 further comprising about 50% by weight of the total compound of an asphaltic residual oil.
- 9. The lubricating compound of claim 2 wherein the hydrocarbon oil is selected from the group consisting of the mineral oils, polybutene synthetic oils and ester synthetic oils.
- 10. The process for the production of a thixotropic lubricating compound having a reversible gel structure comprising the steps of:
 - adding to a reaction vessel significant amounts of a fish oil, a sulfur-modified sperm oil and a microcrystalline wax;

heating the mixture to a temperature of from about. 300°F to about 400°F;

adding to the mixture a significant amount of an oxide of lead; and

cooling the mixture.

- 11. The process of claim 10 comprising the further step of adding a hydrocarbon oil to the mixture in an amount of to 90% by weight of the total resulting composition before cooling the lubricating compound and subsequent to adding the oxide of lead thereto.
- 12. The process of claim 10 comprising the further step of melting the cooled lubricating compound into a hydrocarbon oil wherein the hydrocarbon oil is present in an amount of up to about 90% of the resulting
- 13. The lubricating compound of claim 2 which is further mixed with a volatile solvent such as to render it suitable for application as an aerosol spray.
- 14. The lubricating compound of claim 13 wherein the volatile solvent is selected from the group consisting of the hydrocarbon solvents, chlorinated hydrocarbon solvents, and alcohols.
- 15. A thixotropic lubricating compound having a reversible gel structure comprising the reaction product of significant amounts of a crude fish oil, a sulfurmodified sperm oil, a microcrystalline wax and an amount of an oxide of lead in excess of that amount which would be required to react with the crude fish oil to form a normal lead soap.

16. The lubricating compound of claim 15 further comprising up to about 90 percent by weight of the total compound of a hydrocarbon oil.

- 17. The lubricating compound of claim 15 wherein the crude fish oil is selected from the group consisting of crude tuna oil, herring oil, menhaden oil, sardine oil, cod liver oil, halibut oil, salmon oil and shark oil and the sulfur-modified sperm oil is selected from the group consisting of sulfurized sperm oil and sulfonated sperm
- 18. The lubricating compound of claim 17 wherein the weight ratio of the fish oil to the oxide of lead is from about 1:2 to about 2:1; the weight ratio of the fish oil plus the oxide of lead to the sulfur-modified sperm oil is from about 1:1 to about 4.4:1; and the weight ratio of the microcrystalline wax to the fish oil plus the oxide of lead is from about 1:2 to about 6.5:1.
- 19. The lubricating compound of claim 18 wherein the oxide of lead is litharge.
- 20. The lubricating compound of claim 18 further comprising up to about 90 percent by weight of the total compound of a hydrocarbon oil.
- 21. The lubricating compound of claim 18 wherein when the fish oil is crude tuna oil, the ratio of the fish crocrystalline wax to the fish oil plus litharge is about 55 oil to the oxide of lead is about 1.3:1; the weight ratio of the fish oil plus the oxide of lead to the sulfurmodified sperm oil is about 2.3:1; and the weight ratio of the microcrystalline wax to the fish oil plus litharge is about 1.3:1.
 - 22. The lubricating compound of claim 21 further comprising about 50 percent by weight of the total compound of an asphaltic residual oil.
 - 23. The lubricating compound of claim 16 wherein the hydrocarbon oil is selected from the group consist-65 ing of the mineral oils, polybutene synthetic oils and ester synthetic oils.
 - 24. The lubricating compound of claim 16 which is further mixed with a volatile solvent such as to render it suitable for application as an aerosol spray.

25. The lubricating compound of claim 24 wherein the volatile solvent is selected from the group consisting of the hydrocarbon solvents, chloronated hydrocarbon solvents and alcohols.

26. The process of claim 10 wherein the oxide of lead is added in an amount in excess of that amount which would be required to react with the fish oil present in the lubricating compound to form a normal lead soap.