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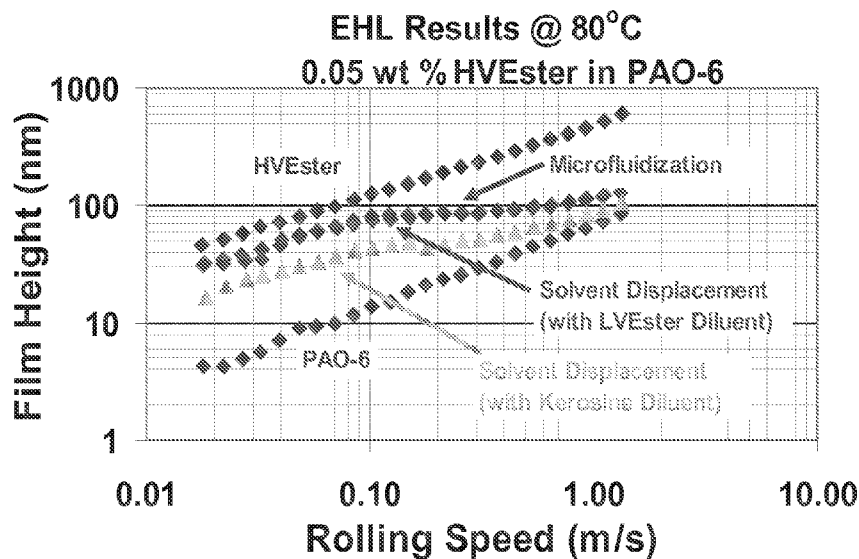


Figure 1

(57) Abstract: This invention provides a method for producing a lubricant composition that is comprised of a continuous phase and a discontinuous phase. The method involves mixing together high viscosity ester, ether or combination thereof, and polar diluent or solvent, to produce a solution. The solution is then added to a low viscosity Group IV base oil to produce a discontinuous phase dispersed throughout a continuous phase.

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A METHOD FOR PRODUCING A TWO PHASE LUBRICANT COMPOSITION

FIELD OF THE INVENTION

[0001] This invention is directed to a method for producing a lubricant composition that is comprised of a continuous phase and a discontinuous phase. More specifically, this invention is directed to a method of producing a two phase lubricant composition by mixing together high viscosity ester, ether or combination thereof and polar solvent to produce a solution, and adding the solution to a low viscosity Group IV base oil to produce a discontinuous phase dispersed throughout a continuous phase.

BACKGROUND OF THE INVENTION

[0002] A particular class of lubricant composition can be characterized as liquid lubricants having at least two distinct liquid phases. Often, these types of lubricants are considered as dispersions, although they are also referred to as emulsions. These types of lubricants are readily identified as having a relatively small quantity of the discontinuous phase, which is comprised of an oil type component and dispersed throughout the continuous oil base oil phase. Since an oil type composition is dispersed through another oil type composition, these lubricants are also referred to as oil-in-oil emulsions.

[0003] Oil-in-oil emulsions can provide substantial wear protection, yet be lower in viscosity relative to standard mineral oil type blended lubricants. Such lubricants can be useful in many applications and are desirable for their superior properties related to low viscosities, improved film thickness, and better lubricating performance.

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[0004] US Patent No. 6,972,275, Forbus, discloses an oil-in-oil emulsion type of lubricant composition. The particular lubricant composition has a continuous phase of a carrier fluid comprised of polyalphaolefins and alkylated aromatics, and a discontinuous phase of a higher viscosity fluid. The carrier fluid and the high viscosity fluid are substantially immiscible and together form a relatively stable emulsion.

[0005] Two phase lubricants such as oil-in-oil emulsions have very good potential for numerous commercial applications. However, additional improvements in overall lubricant quality and performance are desired. Additional wear protection and lower drag are examples of qualities in which improvements are being sought.

SUMMARY OF THE INVENTION

[0006] This invention provides a method for producing a lubricant composition that is comprised of a continuous phase and a discontinuous phase, i.e., a two phase lubricant composition. The lubricant composition that is produced by the method of this invention provides enhanced performance and additional wear protection relative to comparable lubricants. It also provides lower drag performance characteristics relative to comparable lubricants.

[0007] According to one aspect of the invention, there is provided a method for producing a lubricant composition having a discontinuous phase dispersed throughout a continuous phase. The method comprises a step of mixing together high viscosity ester, high viscosity ether or combination thereof, and polar solvent to produce a solution. The high viscosity ester, high viscosity ether, or combination of high viscosity ester and ether, have, independently (i.e., prior to mixing with additional lubricant components) a viscosity of at least 100 cSt at 100°C.

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[0008] At least a portion of the solution is added to a low viscosity Group IV base oil, in which the low viscosity base oil has a viscosity of less than 100 cSt at 100°C to produce the discontinuous phase dispersed throughout the continuous phase. The discontinuous phase is comprised of droplets of the high viscosity ester, with the droplets having a mean average diameter of from 0.1 microns to 20 microns.

[0009] In one embodiment, the solvent has a viscosity of from 1 cSt to 100 cSt at 100°C. Alternatively, the solvent has a viscosity of from 1 cSt to 80 cSt at 100°C.

[0010] The high viscosity ester, high viscosity ether and the low viscosity Group IV base oil are miscible in the solvent. Preferably, the high viscosity ester and high viscosity ether are more highly miscible in the solvent than the low viscosity Group IV base oil.

[0011] In a particular embodiment, the solvent is an aprotic polar composition. Preferably, the solvent has an aniline point of not greater than 45°C. Alternatively or in combination, the solvent has a flash point of at least 150°C.

[0012] In an embodiment, the solvent is a low viscosity ester composition. Preferably, the low viscosity ester solvent is comprised of adipate ester.

[0013] At least a portion of the high viscosity ester composition can be comprised of an ester containing at least one ether linkage. Alternatively, at least a portion of the high viscosity ester composition is comprised of an ester containing no ether linkages.

[0014] In an embodiment, the solution that is added to the base oil contains from 0.1 wt % to 10 wt % solvent, based on total weight of the solution prior to

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mixing with the low viscosity Group IV base oil. Preferably, the solution is added to the low viscosity Group IV base oil at a rate of at least 0.1 ml/min. It is desirable that the solution be added to the low viscosity Group IV base oil at a temperature of from 20°C to 80°C.

[0015] The low viscosity Group IV base oil that is used as a component of the lubricant compositions has, independently, a viscosity of from 1 cSt to 100 cSt at 100°C.

[0016] The lubricant composition can be microfluidized following production of the dispersed phase. Such a step is considered optional to the overall process.

BRIEF DESCRIPTION OF THE DRAWINGS

[0017] Figure 1. EHL Results for Solvent Displacement Emulsions Compared to a Microfluidized Emulsion.

[0018] Figure 2. EHL Film Thickness of 0.5 wt% Dispersions of PTE in PAO-8 in Comparison to Pure PAO-8 and Pure PTE.

[0019] Figure 3. EHL Film Thickness of 0.5 wt% Dispersions of PTE in PAO-8 in Comparison to Pure PAO-8 and Pure PTE.

[0020] Figure 4. EHL Film Thickness of 0.5% Complex Ester Dispersions in PAO-8 in Comparison to Pure PAO-8 and Pure Complex Ester.

[0021] Figure 5. EHL Film Thickness of 0.5% Complex Ester Dispersions in PAO-8 in Comparison to Pure PAO-8 and Pure Complex Ester.

[0022] Figure 6. Friction Coefficient of Microfluidized 0.5% Complex Ester in PAO-8 Compared to Pure PAO-8 and Pure Complex Ester.

DETAILED DESCRIPTION OF THE INVENTION

Introduction

[0023] This invention provides a method for producing a lubricant composition that is comprised of a continuous phase and a discontinuous phase. The method involves mixing together high viscosity ester, ether or combination thereof, and polar diluent or solvent, to produce a solution. The solution is then added to a low viscosity Group IV base oil to produce a discontinuous phase dispersed throughout a continuous phase.

[0024] The high viscosity ester, high viscosity ether, and low viscosity Group IV base oil are miscible in the polar solvent. However, the high viscosity ester and high viscosity ether used in the process of this invention are substantially immiscible in the low viscosity Group IV base oil. The use of the polar solvent enables formation of the discontinuous phase at a reduced energy level compared to previous methods. Lubricant performance can be enhanced further by carrying out high shear homogenization such as by microfluidization. The resulting two phase lubricant composition demonstrates substantial elastohydrodynamic film thickness, reduced droplet size in the discontinuous phase and is less sensitive to changes in carrier oil. The lubricant composition also has a prolonged shelf life.

Continuous Phase Base Oil (low viscosity Group IV base oil)

[0025] The lubricant composition produced according to this invention is comprised of a continuous phase base oil and a discontinuous phase oil. The continuous phase base oil has relatively low viscosity, i.e., lower than that of the discontinuous phase oil. For example, the Group IV continuous phase base oil has a viscosity at 100°C of at least 10 cSt, or at least 30 cSt, or at least 50 cSt or at least 70 cSt lower than that of the discontinuous phase oil.

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[0026] The continuous phase base oil is comprised of Group IV base oil comprised of one or more Group IV base stocks. The terms “base oil” and “base stock” as referred to herein are to be considered consistent with the definitions as also stated in APPENDIX E—API BASE OIL INTERCHANGEABILITY GUIDELINES FOR PASSENGER CAR MOTOR OILS AND DIESEL ENGINE OILS, July 2009 Version. According to Appendix E, base oil is the base stock or blend of base stocks used in an API-licensed oil. Base stock is a lubricant component that is produced by a single manufacturer to the same specifications (independent of feed source or manufacturer’s location); that meets the same manufacturer’s specification; and that is identified by a unique formula, product identification number, or both.

[0027] The continuous phase base oil has, independently, a viscosity of from 1 cSt to 100 cSt at 100°C. The term independently means that the viscosity of the continuous phase base oil is determined after blending all of the individual base stocks together that comprise the continuous base oil phase, and prior to blending with the discontinuous phase base oil of relatively higher viscosity. Preferably, the continuous phase base oil has, independently, a viscosity of not greater than 80 cSt at 100°C, alternatively, not greater than 50 cSt at 100°C or not greater than 30 cSt at 100°C. Exemplary ranges include from 1 cSt to 80 cSt at 100°C, from 1 cSt to 50 cSt at 100°C and from 1 cSt to 30 cSt at 100°C.

[0028] Group IV base stocks are polyalphaolefins (PAOs). PAOs can be obtained by polymerizing at least one monomer, e.g., 1-olefin, in the presence of hydrogen and a catalyst composition. Alpha-olefins suitable for use in the preparation of the PAOs can contain from 2 to about 30, preferably from 2 to 20, carbon atoms, and more preferably from about 6 to about 12 carbon atoms. Non-limiting examples of such alpha-olefins include ethylene, propylene, 2-methylpropene, 1-butene, 3-methyl-1-butene, 1-pentene, 4-methyl-1-pentene,

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1-hexene, 1-heptene, 1-octene, 1-nonene, 1-decene, 1-undecene, 1-dodecene, 1-tridecene, 1-tetradecene, 1-pentadecene, 1-hexadecene, 1-heptadecene, 1-octadecene, 1-nonadecene, and 1-eicosene, including mixtures of at least two of the alpha-olefins. Preferred alpha-olefins for use herein are 1-octene, 1-decene and 1-dodecene, including mixtures thereof.

[0029] Specifically, the PAOs that can be used according to this invention can be produced by polymerization of olefin feed in the presence of a catalyst such as AlCl_3 , BF_3 , or promoted AlCl_3 , BF_3 . Processes for the production of such PAOs are disclosed, for example, in the following patents: U.S. Pat. Nos. 3,149,178; 3,382,291; 3,742,082; 3,769,363; 3,780,128; 4,172,855 and 4,956,122, which are fully incorporated by reference. Additional PAOs are also discussed in: Will, J. G. *Lubrication Fundamentals*, Marcel Dekker: New York, 1980. Subsequent to polymerization, the PAO lubricant range products are typically hydrogenated in order to reduce the residual unsaturation, generally to a level of greater than 90% of hydrogenation.

[0030] Low viscosity PAOs that can be used according to the invention can be produced by polymerization of an alpha-olefin in the presence of a polymerization catalyst such as Friedel-Crafts catalysts. These include, for example, boron trichloride, aluminum trichloride, or boron trifluoride, promoted with water, with alcohols such as ethanol, propanol, or butanol, with carboxylic acids, or with esters such as ethyl acetate or ethyl propionate or ether such as diethyl ether, diisopropyl ether, etc. (See for example, the methods disclosed by U.S. Pat. No. 4,149,178 or 3,382,291.) Other descriptions of PAO synthesis are found in the following patents: U.S. Pat. No. 3,742,082 (Brennan); U.S. Pat. No. 3,769,363 (Brennan); U.S. Pat. No. 3,876,720 (Heilman); U.S. Pat. No. 4,239,930 (Allphin); U.S. Pat. No. 4,367,352 (Watts); U.S. Pat. No. 4,413,156 (Watts); U.S. Pat. No.

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4,434,408 (Larkin); U.S. Pat. No. 4,910,355 (Shubkin); U.S. Pat. No. 4,956,122 (Watts); and U.S. Pat. No. 5,068,487 (Theriot).

[0031] Another class of PAOs that can be incorporated as a part of this invention can be prepared by the action of a supported, reduced chromium catalyst with an alpha-olefin monomer. Such PAOs are described in U.S. Pat. No. 4,827,073 (Wu); U.S. Pat. No. 4,827,064 (Wu); U.S. Pat. No. 4,967,032 (Ho et al.); U.S. Pat. No. 4,926,004 (Pelrine et al.); and U.S. Pat. No. 4,914,254 (Pelrine). Commercially available PAOs include SpectraSyn™ 2, 4, 5, 6, 8, 10, 40 and 100. (ExxonMobil Chemical Company, Houston, Tex.).

[0032] PAOs made using metallocene catalyst systems can also be used according to this invention. Examples are described in U.S. Pat. No. 6,706,828 (equivalent to US 2004/0147693), where PAOs having KV100s of greater than 1,000 cSt are produced from meso-forms of certain metallocene catalysts under high hydrogen pressure with methyl alumoxane as a activator.

[0033] PAOs, such as polydecene, using various metallocene catalysts can also be incorporated into the lubricating composition of this invention. Examples of how such PAOs can be produced are described, for example, in WO 96/23751, EP 0 613 873, U.S. Pat. No. 5,688,887, U.S. Pat. No. 6,043,401, WO 03/020856 (equivalent to US 2003/0055184), U.S. Pat. No. 5,087,788, U.S. Pat. No. 6,414,090, U.S. Pat. No. 6,414,091, U.S. Pat. No. 4,704,491 U.S. Pat. No. 6,133,209, and U.S. Pat. No. 6,713,438.

[0034] In one embodiment of the invention, the polyolefin base oil component of this invention has a M_w (weight average molecular weight) of about 200,000 or less, preferably from about 250 to 150,000, alternatively from about 280 to 100,000, or from about 300 to about 75,000 g/mol.

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[0035] The polyolefin base oil component of this invention can have a molecular weight distribution (MWD) of greater than 1. MWD is defined as the ratio of weight-averaged MW to number-averaged MW (M_w/M_n), which is preferably determined by gel permeation chromatography (GPC) using polystyrene standards, as described in *Principles of Polymer Systems*, Fourth Edition, Ferdinand Rodrigues, Chapter 6, McGraw-Hill Book. An example of GPC solvent is HPLC Grade tetrahydrofuran, uninhibited, with the procedure being carried out at a column temperature of 30°C, a flow rate of 1 ml/min, and a sample concentration of 1 wt%, with a Column Set being a Phenogel 500 A, Linear, 10E6A. In one embodiment, the MWD is less than 5, preferably less than 4, preferably less than 3, preferably less than 2.5, preferably less than 2. Alternatively, polyolefin base oil component has a M_w/M_n of from 1 to 3.5, alternatively from 1 to 2.5.

[0036] In one embodiment, the polyolefin base oil component has an unimodal M_w/M_n determined by GPC. In another embodiment, the polyolefin base oil component has a multi-modal molecular weight distribution, where the MWD can be greater than 5. In another aspect, the polyolefin base oil component has a shoulder peak either before or after, or both before and after the major unimodal distribution. In this case, the MWD can be broad (>5) or narrow (<5 or <3 or <2), depending on the amount and size of the shoulder.

[0037] For many applications when superior shear stability, thermal stability or thermal/oxidative stability is preferred, it is preferable to have the polyolefins made with the narrowest possible MWD. PAO fluids with different viscosities, but made from the same feeds or catalysts, usually have different MWDs. In other words, MWDs of PAO fluids are dependent on fluid viscosity. Usually, lower viscosity fluids have narrower MWDs (smaller MWD value) and higher viscosity fluids have broader MWDs (larger MWD value). For a polyolefin base

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oil component with 100°C KV of less than 100 cSt, or not greater than 80 cSt, or not greater than 50 cSt, or not greater than 30 cSt, the MWD of is preferably less than 2.5, alternatively less than 2.3.

[0038] M_w and M_n are also preferably measured by GPC method using a column for medium- to low-molecular weight polymers. Preferably the GPC method is carried out with tetrahydrofuran as solvent and polystyrene as calibration standard, as described above for MWD determination.

[0039] In a preferred embodiment of this invention, the polyolefin base oil component has a pour point of less than 25°C (as measured by ASTM D 97), preferably less than 0°C, preferably less than -10°C, preferably less than -20°C, preferably less than -25°C, preferably less than -30°C, preferably less than -35°C, preferably less than -40°C, preferably less than -55°C, preferably from -10°C to -80°C, preferably from -15°C to -70°C.

[0040] Preferably, the polyolefin base oil component has a peak melting point (T_m) of 0°C or less, and preferably have no measurable T_m . "No measurable T_m " is defined to be when there is no clear melting as observed by heat absorption in the DSC heating cycle measurement. Usually the amount of heat absorption is less than 20 J/g. It is preferred to have the heat release of less than 10 J/g, preferred less than 5 J/g, more preferred less than 1 J/g. Usually, it is preferred to have lower melting temperature, preferably below 0°C, more preferably below -10°C, more preferably below -20°C, more preferably below -30°C, more preferably below -40°C, most preferably no clear melting peak in DSC.

[0041] Peak melting point (T_m), crystallization temperature (T_c), heat of fusion and degree of crystallinity (also referred to as % crystallinity) can be determined using the following procedure. Differential scanning calorimetric (DSC) data is obtained using a TA Instruments model 2920 machine. Samples weighing

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approximately 7-10 mg are sealed in aluminum sample pans. The DSC data can be recorded by first cooling the sample to -100°C , and then gradually heating to 30°C at a rate of $10^{\circ}\text{C}/\text{minute}$. The sample can be kept at 30°C for 5 minutes before a second cooling-heating cycle is applied. Both the first and second cycle thermal events should be recorded. Areas under the curves are preferably measured and used to determine the heat of fusion and the degree of crystallinity. Additional details of such procedure is described in US Patent Pub. No. 2009/0036725.

[0042] In one embodiment of the invention, the polyolefin base oil component is preferred to have no appreciable cold crystallization in DSC measurement. During the heating cycle for the DSC method as described above, the PAO may crystallize if it has any crystallizable fraction. This cold crystallization can be observed on the DSC curve as a distinct region of heat release. The extent of the crystallization can be measured by the amount of heat release. Higher amount of heat release at lower temperature means higher degree of poor low temperature product. The cold crystallization is usually less desirable, as it may mean that the fluid may have very poor low temperature properties – not suitable for high performance application. It is preferred to have less than 20 j/g of heat release for this type of cold crystallization, preferred less than 10 j/g, less than 5 j/g and less than 1 j/g, most preferably to have no observable heat release due to cold crystallization during DSC heating cycle.

[0043] In another preferred embodiment, the polyolefin base oil component will have a viscosity index (VI) of greater than 60, preferably greater than 100, more preferably greater than 120, preferably at least 160 and more preferably at least 180. VI is determined according to ASTM Method D 2270-93 [1998]. VI of a fluid is usually dependent on the viscosity, feed composition and method of preparation. Higher viscosity fluid of the same feed composition usually has

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higher VI. The typical VI range for fluids made from C_3 or C_4 or C_5 linear alpha-olefin (LAO) will typically be from 65 to 250. Typical VI range for fluids made from C_6 or C_7 will be from 100 to 300, depending on fluid viscosity. Typical VI range for fluids made from C_8 to C_{14} LAO, such as 1-octene, 1-nonene, 1-decene or 1-undecene or 1-dodecene, 1-tetra-decene, are from 120 to >450, depending on viscosity. More specifically, the VI range for fluids made from 1-decene or 1-decene equivalent feeds are from about 100 to about 500, preferably from about 120 to about 400. Two or three or more alpha-olefins can be used as feeds, such as combination of C_3+C_{10} , C_3+C_{14} , C_3+C_{16} , C_3+C_{18} , C_4+C_8 , C_4+C_{12} , C_4+C_{16} , $C_3+C_4+C_8$, $C_3+C_4+C_{12}$, $C_4+C_{10}+C_{12}$, $C_4+C_{10}+C_{14}$, C_6+C_{12} , $C_6+C_{12}+C_{14}$, $C_4+C_6+C_{10}+C_{14}$, $C_4+C_6+C_8+C_{10}+C_{12}+C_{14}+C_{16}+C_{18}$, etc. The product VI depends on the fluid viscosity and also on the choice of feed olefin composition. For the most demanding lubricant applications, it is better to use fluids with higher VI.

[0044] In another embodiment, it is preferable that the PAO base oil does not contain a significant amount of very light fraction. These light fractions contribute to high volatility, unstable viscosity, poor oxidative and thermal stability. They are usually removed in the final product. It is generally preferable to have less than 5 wt % of the polyolefin base oil with C_{20} or lower carbon numbers, more preferably less than 10 wt % of the polyolefin base oil with C_{24} or lower carbon numbers or more preferably less than 15 wt % of the polyolefin base oil with C_{26} or lower carbon numbers. It is preferable to have less than 3 wt % of the polyolefin base oil with C_{20} or lower carbon numbers, more preferably less than 5 wt % of the polyolefin base oil with C_{24} or lower carbon numbers or more preferably less than 8 wt % of the polyolefin base oil with C_{26} or lower carbon numbers. It is preferable to have less than 2 wt % of the polyolefin base oil with C_{20} or lower carbon numbers, more preferably less than 3 wt % of the polyolefin base oil with C_{24} or lower carbon numbers or more preferably less than 5 wt % of

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the polyolefin base oil with C₂₆ or lower carbon numbers. Also, the lower the amount of any of these light hydrocarbons, the better the fluid property of the polyolefin base oil as can be determined by Noack volatility testing (ASTM D5800).

[0045] In another embodiment, it is preferable that the PAO base oil does not contain a significant amount of a high molecular weight fraction. Such PAOs can be made by removing the high MW fraction or by using preferred metallocene catalysts (referred to as mPAO). Preferably, the PAO has not more than 5.0 wt% of polymer having a molecular weight of greater than 45,000 Daltons. Additionally or alternately, the amount of the PAO that has a molecular weight greater than 45,000 Daltons is not more than 1.5 wt%, or not more than 0.10 wt%. Additionally or alternately, the amount of the PAO that has a molecular weight greater than 60,000 Daltons is not more than 0.5 wt%, or not more than 0.20 wt%, or not more than 0.1 wt%. The mass fractions at molecular weights of 45,000 and 60,000 can be determined by GPC, as described above.

[0046] In general, Noack volatility is a strong function of fluid viscosity. Lower viscosity fluid usually has higher volatility and higher viscosity fluid has lower volatility. Preferably, the polyolefin base oil has a Noack volatility of less than 30 wt %, preferably less than 25 wt %, preferably less than 10 wt %, preferably less than 5 wt %, preferably less than 1 wt %, and preferably less than 0.5 wt %.

[0047] In another embodiment, the polyolefin base oil has a dielectric constant of 3 or less, usually 2.5 or less (1 kHz at 23°C, as determined by ASTM D 924).

[0048] In another embodiment, the polyolefin base oil can have a specific gravity of 0.6 to 0.9 g/cm³, preferably 0.7 to 0.88 g/cm³.

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[0049] In another embodiment, the PAO's produced directly from the oligomerization or polymerization process are unsaturated olefins. The amount of unsaturation can be quantitatively measured by bromine number measurement according to the ASTM D 1159, or by proton or carbon-13 NMR. Proton NMR spectroscopic analysis can also differentiate and quantify the types of olefinic unsaturation: vinylidene, 1,2-disubstituted, trisubstituted, or vinyl. Carbon-13 NMR spectroscopy can confirm the olefin distribution calculated from the proton spectrum.

[0050] Both proton and carbon-13 NMR spectroscopy can quantify the extent of short chain branching (SCB) in the olefin oligomer, although carbon-13 NMR can provide greater specificity with respect to branch lengths. In the proton spectrum, the SCB branch methyl resonances fall in the 1.05-0.7 ppm range. SCBs of sufficiently different length will give methyl peaks that are distinct enough to be integrated separately or deconvoluted to provide a branch length distribution. The remaining methylene and methine signals resonate in the 3.0-1.05 ppm range. In order to relate the integrals to CH, CH₂, and CH₃ concentrations, each integral must be corrected for the proton multiplicity. The methyl integral is divided by three to derive the number of methyl groups; the remaining aliphatic integral is assumed to comprise one CH signal for each methyl group, with the remaining integral as CH₂ signal. The ratio of CH₃/(CH+CH₂+CH₃) gives the methyl group concentration.

[0051] Similar logic applies to the carbon-13 NMR analysis, with the exception that no proton multiplicity corrections need be made. Furthermore, the enhanced spectral/structural resolution of ¹³C NMR vis a vis ¹H NMR allows differentiation of ions according to branch lengths. Typically, the methyl resonances can be integrated separately to give branch concentrations for methyls

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(20.5-15 ppm), propyls (15-14.3 ppm), butyl-and-longer branches (14.3-13.9 ppm), and ethyls (13.9-7 ppm).

[0052] Olefin analysis is readily performed by proton NMR, with the olefinic signal between 5.9 and 4.7 ppm subdivided according to the alkyl substitution pattern of the olefin. Vinyl group CH protons resonate between 5.9-5.7 ppm, and the vinyl CH₂ protons between 5.3 and 4.85 ppm. 1,2-disubstituted olefinic protons resonate in the 5.5-5.3 ppm range. The trisubstituted olefin peaks overlap the vinyl CH₂ peaks in the 5.3-4.85 ppm region; the vinyl contributions to this region are removed by subtraction based on twice the vinyl CH integral. The 1,1-disubstituted- or vinylidene-olefins resonate in the 4.85-4.6 ppm region. The olefinic resonances, once corrected for the proton multiplicities can be normalized to give a mole-percentage olefin distribution, or compared to the multiplicity-corrected aliphatic region (as was described above for the methyl analysis) to give fractional concentrations (e.g. olefins per 100 carbons).

[0053] Generally, the amount of unsaturation strongly depends on fluid viscosity or fluid molecular weight. Lower viscosity fluid has higher degree of unsaturation and higher bromine number. Higher viscosity fluid has lower degree of unsaturation and lower bromine number. If a large amount of hydrogen or high hydrogen pressure is applied during the polymerization step, the bromine number can be lower than without the hydrogen presence. Typically, for PAO produced from 1-decene or other suitable LAOs, the as-synthesized PAO will have bromine number of from 80 to less than 1, but greater than 0, preferably from about 40 to about 0.01, preferably from about 20 to about 0.5, depending on fluid viscosity.

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Discontinuous Phase (high viscosity ester/ether)

a. High Viscosity Ester and/or Ether Composition

[0054] The discontinuous phase is represented by droplets dispersed throughout the continuous phase. The droplets are relatively evenly dispersed throughout the continuous phase and remain so for very long periods of time. The droplets are of a size to resist rapid coalescence, this providing for a stable dispersion of droplets. The mean number average droplet size (as determined by laser light scattering) is not greater than 20 microns, typically from 0.01 micron to 20 microns. The droplets can also be dispersed throughout the continuous phase at a mean number average droplet size of not greater than 10 microns or 5 microns.

[0055] The discontinuous phase is comprised of an ester and/or ether composition, independently higher in viscosity than the continuous phase base oil. Preferably, the discontinuous phase has, independently, a viscosity of greater than 100 cSt at 100°C. As with the continuous phase base oil, independently means that the viscosity of the discontinuous phase is determined after blending all of the individual base stocks together that comprise the discontinuous phase, and prior to blending with the continuous phase base oil of relatively lower viscosity. Preferably, the discontinuous phase base oil has, independently, a viscosity of at least 120 cSt at 100°C, or at least 140 cSt at 100°C or at least 160 cSt at 100°C. Exemplary ranges include from greater than 100 cSt to 6,000 cSt at 100°C, from 120 cSt to 4,000 cSt at 100°C and from 140 cSt to 3,000 cSt at 100°C.

b. High Viscosity Ester Composition

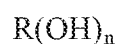
[0056] The high viscosity ester composition is the reaction product of at least one carboxylic acid or anhydride and at least one alcohol. In one embodiment, the

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high viscosity ester composition is comprised of a complex ester. A complex ester is considered a reaction product of a polyol, a polybasic acid or anhydride, and a mono-alcohol.

[0057] In a particular embodiment, the complex ester is a reaction product of:

- a. a polyhydroxyl compound represented by the general formula:



wherein R is an aliphatic or cyclo-aliphatic hydrocarbyl group and n is at least 2, with the hydrocarbyl group preferably containing from about 2 to 20 carbon atoms;

- b. a polybasic acid or an anhydride of a polybasic acid, preferably with a ratio of equivalents of the polybasic acid to equivalents of alcohol of the polyhydroxyl compound in the range of from about 1.6:1 to 2:1; and
- c. a monohydric alcohol, preferably at a ratio of equivalents of the monohydric alcohol to equivalents of the polybasic acid in the range of from about 0.8:1 to 1.2:1.

[0058] Among the polyols (i.e., polyhydroxyl compounds) which can be reacted to produce the complex ester are those represented by the general formula:



wherein R is an aliphatic or cyclo-aliphatic hydrocarbyl group (preferably an alkyl) and n is at least 2. The hydrocarbyl group can contain from about 2 to about 20 or more carbon atoms, and the hydrocarbyl group can also contain substituents such as chlorine, nitrogen and/or oxygen atoms.

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[0059] The polyhydroxyl compounds preferably include no oxyalkylene groups and, thus, the polyhydroxyl compounds exclude compounds such as polyetherpolyols. The number of carbon atoms (i.e., carbon number, wherein the term carbon number as used throughout this application refers to the total number of carbon atoms in either the acid or alcohol as the case may be) and number of hydroxy groups (i.e., hydroxyl number) contained in the polyhydroxyl compound used to form the carboxylic esters may vary over a wide range.

[0060] The following alcohols are particularly useful as polyols: neopentyl glycol, trimethylolethane, trimethylolpropane, trimethylolbutane, mono-pentaerythritol, technical grade pentaerythritol, and di-pentaerythritol. Particularly preferred alcohols are technical grade (e.g., approximately 88% mono-, 10% di- and 1-2% tri-pentaerythritol) pentaerythritol, monopentaerythritol, di-pentaerythritol, and trimethylolpropane.

[0061] Polybasic or polycarboxylic acids that can be used to produce the complex ester include one or more of C_2 to C_{16} diacids. Examples include, but are not limited to adipic, azelaic, sebacic and dodecanedioic acids.

[0062] Anhydrides of polybasic acids can be used in place of the polybasic acids to produce the complex esters. Examples include, but are not limited to, succinic anhydride, glutaric anhydride, adipic anhydride, maleic anhydride, phthalic anhydride, nadic anhydride, methyl nadic anhydride, hexahydrophthalic anhydride, and mixed anhydrides of polybasic acids.

[0063] Among the alcohols which can be reacted with the diacid and polyol are, by way of example, any one or more of C_5 to C_{13} branched and/or linear monohydric alcohol (mono-alcohol) selected from the group consisting of isopentyl alcohol, n-pentyl alcohol, isohexyl alcohol, n-hexyl alcohol, isoheptyl alcohol, n-heptyl alcohol, iso-octyl alcohol (e.g., 2-ethyl hexanol or iso-octyl

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alcohol), n-octyl alcohol, iso-nonyl alcohol, n-nonyl alcohol, isodecyl alcohol, and n-decyl alcohol. Preferably, at least one linear monohydric alcohol is present at up to 20 mole %, based on the total amount of monohydric alcohol.

[0064] An example of a particular class of monohydric alcohol that can be used to produce the complex ester is oxo alcohol. Oxo alcohols are manufactured by a process in which propylene and other olefins are oligomerized over a catalyst (e.g., a phosphoric acid on Kieselguhr clay) and then distilled to achieve various unsaturated (olefinic) streams largely comprising a single carbon number. These streams can then be reacted under hydroformylation conditions using a cobalt carbonyl catalyst with synthesis gas (carbon monoxide and hydrogen) so as to produce a multi-isomer mix of aldehydes/alcohols. The mix of aldehydes/alcohols can then be introduced to a hydrogenation reactor and hydrogenated to a mixture of branched alcohols comprising mostly alcohols of one carbon greater than the number of carbons in the feed olefin stream.

[0065] Examples of branched oxo alcohols include monohydric oxo alcohols which have a carbon number in the range of from about C₅ to C₁₃. A particular oxo alcohol includes iso-octyl alcohol formed from the cobalt oxo process and 2-ethylhexanol which is formed from the rhodium oxo process.

[0066] The term “iso” is meant to convey a multiple isomer product made by the oxo process. It is desirable to have a branched oxo alcohol comprising multiple isomers, preferably more than 3 isomers, most preferably more than 5 isomers.

[0067] Branched oxo alcohols can be produced in the so-called “oxo” process by hydroformylation of branched C₄ to C₁₂ olefin fractions to a corresponding branched C₅ to C₁₃ alcohol/aldehyde-containing oxonation product. In the process for forming oxo alcohols it is desirable to form an alcohol/aldehyde intermediate

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from the oxonation product followed by conversion of the crude oxo alcohol/aldehyde product to an all oxo alcohol product.

[0068] The production of branched oxo alcohols from the cobalt catalyzed hydroformylation of an olefinic feedstream preferably comprises the following steps:

- i. hydroformylating an olefinic feedstream by reaction with carbon monoxide and hydrogen (i.e., synthesis gas) in the presence of a hydroformylation catalyst under reaction conditions that promote the formation of an alcohol/aldehyde-rich crude reaction product;
- ii. demetalling the alcohol/aldehyde-rich crude reaction product to recover therefrom the hydroformylation catalyst and a substantially catalyst-free, alcohol/aldehyde-rich crude reaction product; and
- iii. hydrogenating the alcohol/aldehyde-rich crude reaction product in the presence of a hydrogenation catalyst (e.g., massive nickel catalyst) to produce an alcohol-rich reaction product.

[0069] The olefinic feedstream is comprised of at least one C₄ to C₁₂ olefin, more preferably at least one branched C₇ to C₉ olefin. Moreover, the olefinic feedstream is preferably a branched olefin, although a linear olefin which is capable of producing all branched oxo alcohols can also be used. The hydroformylation and subsequent hydrogenation in the presence of an alcohol-forming catalyst, is capable of producing branched C₅ to C₁₃ alcohols, more preferably branched C₈ alcohol, branched C₉ alcohol, and isodecyl alcohol. Each of the branched oxo C₅ to C₁₃ alcohols formed by the oxo process typically

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comprises, for example, a mixture of branched oxo alcohol isomers alcohol comprises a mixture of 3,5-dimethyl hexanol, 4,5-dimethyl hexanol, 3,4-dimethyl hexanol, 5-methyl heptanol, 4-methyl heptanol and a mixture of other methyl heptanols and dimethyl hexanols. Any type of catalyst capable of converting oxo aldehydes to oxo alcohols can be used to produce the complex ester.

[0070] In an alternative embodiment, complex esters used as the high viscosity ester of this invention refers to esters formed from the reaction of three or more of the following compounds:

- i. Monohydric aliphatic alcohols
- ii. Monobasic aliphatic acids
- iii. Aliphatic glycols or polyglycols
- iv. Polyhydric aliphatic alcohols
- v. Dibasic aliphatic acids
- vi. Polybasic aliphatic acids

where at least one polyfunctional alcohol and at least one polyfunctional acid are employed.

[0071] Examples of the above complex esters include:

Glycol centered complex esters; i.e., esters having a chain exemplified as monohydric alcohol-dibasic acid-(glycol-dibasic acid) x-monohydric alcohol, wherein x is a number greater than 0, preferably about 1 to about 6;

Dibasic acid centered complex esters; i.e., esters having a chain structure which may be exemplified as monobasic acid-glycol-(dibasic acid-

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glycol) x-monobasic acid, wherein x is a number greater than 0, preferably about 1 to about 6;

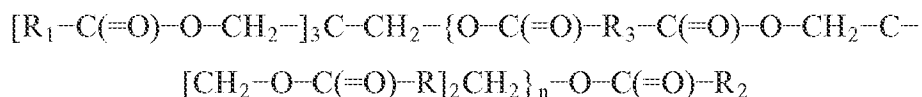
Alcohol acid terminated complex esters; i.e., esters having a chain structure which may be exemplified as monobasic acid-(glycol-dibasic acid) x-monohydric alcohol, wherein x is a number greater than 0, preferably about 1 to about 6; and

Dibasic acid centered complex esters; i.e. esters having a chain structure which may be exemplified as mono-basic acid-polyol-(dibasic acid)-polyol-monobasic acid.

[0072] Preparation of complex esters is disclosed in U.S. Pat. Nos. 2,575,195, 2,575,196, and 3,016,353 and 4,440,657. Generally, the monohydric aliphatic alcohols used in the preparation of these esters will have from about 1 to about 18, and preferably about 4 to about 13 carbon atoms in the molecule and the same may have a straight or branched chain structure. The polyhydric aliphatic alcohols which may be used to prepare esters of this type generally will have from about 4 to about 25 and preferably about 5 to about 20 carbon atoms per molecule and the same may contain ether linkages. The aliphatic glycols or polyglycols may contain from about 2 to about 70 and preferably from about 2 to about 18 carbon atoms per molecule and also may contain ether linkages. Monobasic aliphatic acids which may be used to prepare these esters will generally contain from about 2 to about 22, and preferably from about 4 to about 12 carbon atoms and these materials may have either straight or branched chain structures. The dibasic acids which may be used in the preparation of the complex esters will have from about 2 to about 25, and preferably about 4 to about 14 carbon atoms in the molecule. The polybasic aliphatic acids will contain from about 3 to about 30, and preferably about 4 to about 14 carbon atoms in the molecule.

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[0073] An example of a preferred ester is represented by the formula:



wherein

R_1 and R_2 are independently from 2 to 22 carbons, which can be straight chained or branched, and can further include aliphatic or aromatic rings,

R_3 is from 2 to 25 carbons, and can be straight chained or branched, and can further include aliphatic or aromatic rings, and

n is an integer from 1 to 9.

c. High Viscosity Ether Composition

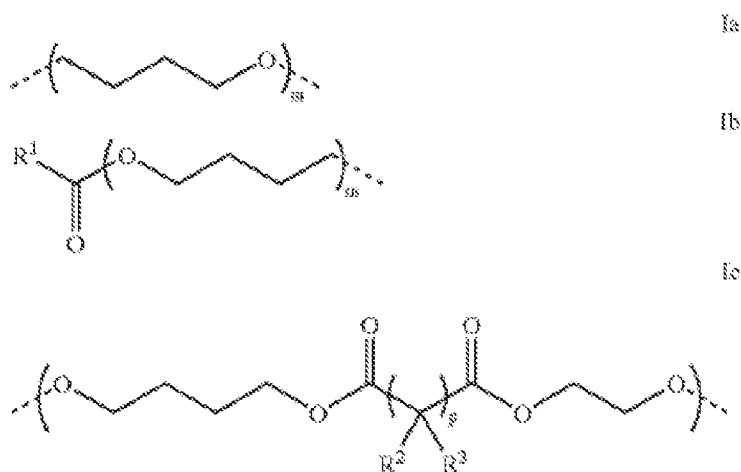
[0074] High viscosity esters such as polyethers and derivatives thereof can be used to form the discontinuous phase of the lubricant composition. Examples of such poly ethers include, but are not limited to, polymers or oligomers containing a plurality of ether moieties such as polyalkylene glycols (e.g., polypropylene glycol and polyethylene glycol) and their corresponding monoethers, diethers, monoesters, and diesters. Additional examples include polyethers derived from the polymerization of cyclic ethers such as epoxides and oxiranes, including tetrahydrofuran.

d. High Viscosity Ester with Ether Linkages

[0075] High viscosity esters having ether linkages can also be used to produce the discontinuous phase of the lubricant according to this invention. Examples of high viscosity esters having ether linkages include poly-tetrahydrofuran (p-THF) ester fluids. These fluids can be made by the condensation reaction between

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p-THF and dibasic carboxylic acids to yield crosslinked p-THF products which are further reacted with monobasic carboxylic acids to endcap the terminal hydroxyl groups in a second condensation reaction. The resulting p-THF ester fluid may be described as comprising one or more of the components depicted in formulas Ia, Ib, and Ic.



wherein

R^1 , R^2 and R^3 are each, independently, hydrogen or any substituted or unsubstituted C_1 to C_{30} , preferably C_1 to C_{20} , alkyl, aryl, aralkyl, alkoxy or aryloxy group, including, but not limited to, those having the groups selected from methyl, ethyl, n-propyl, isopropyl, n-butyl, t-butyl, phenyl, and benzyl; and

m and p are each, independently, any integer of 1 or more, preferably not greater than 30 such as not greater than 20.

Polar Solvent or Diluent

[0076] The solvent used to produce the two phase lubricant composition according to this invention is a polar solvent, particularly a polar hydrocarbon

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solvent. A hydrocarbon is considered to be a compound that is comprised of at least one carbon and at least one hydrogen atom. Polar hydrocarbon solvents can have characteristics of one or more of a large dipole moment and high dielectric constant.

[0077] In an embodiment, the solvent will have a dipole moment of at least 1.5 D, preferably at least 1.6 D, and more preferably at least 1.8 D. In an alternate embodiment, the solvent will have a dielectric constant (k) of at least 5, preferably at least 6, more preferably at least 7. Each of the measurements indicated are measurements under standard temperature and pressure conditions, i.e., 20°C and 1 atmosphere.

[0078] In one embodiment, the polar solvent is an aprotic composition. Aprotic solvents refer to compounds that do not contain dissociable H⁺, or that do not donate a proton (H⁺), but have a large bond dipole. Typically, this large bond dipole is generated by a multiple bond between carbon and either oxygen or nitrogen. Preferably, the aprotic solvents are dipolar aprotic compounds that contain a carbon-oxygen double bond. Examples of aprotic solvents include, but are not limited to, esters, ketones, amides, nitriles and sulfoxides. Preferred aprotic solvents are esters.

[0079] The solvent can be made of a mixture of compounds. In such a case, not every compound in the mixture has to individually meet the desired characteristics of polarity such as dipole moment and dielectric constant. However, in the case that the solvent is comprised of a mixture of compounds, the overall mixture should exhibit the desired polarity characteristics. For example, the mixture of components should exhibit the desired characteristics of one or more desired dipole moment and dielectric constant.

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[0080] In an embodiment, solvent composition has a boiling point at 1 atmosphere of at least 50°C are preferred. Higher boiling point flushing fluids such as those have a boiling point of at least 70°C, or at least 90°C, or at least 120°C can be effectively used. In the case of the solvent comprising a mixture of compounds, the boiling point of the mixture refers to final boiling point as determined by testing method ASTM D 86.

[0081] The solvent used according to this invention is miscible with the high viscosity ester, high viscosity ether and the low viscosity Group IV base oil. Preferably, the high viscosity ester and high viscosity ether are more highly miscible in the solvent than the low viscosity Group IV base oil.

[0082] In one embodiment the the solvent has a viscosity of less than 100 cSt at 100°C, alternatively less than 80 cSt at 100°C or less than 60 cSt at 100°C or less than 40 cSt at 100°C or less than 20 cSt at 100°C or less than 10 cSt at 100°C. For example, the solvent can have a viscosity of from 1 cSt to 100 cSt at 100°C or from 1 cSt to 80 cSt at 100°C or to 60 cSt at 100°C or to 40 cSt at 100°C or to 20 cSt at 100°C or to 10 cSt at 100°C.

[0083] The solvent can have a low aniline point. In one embodiment, the solvent has an aniline point of not greater than 45°C. Alternatively, the solvent has an aniline point of not greater than 35°C or greater than 25°C. Aniline point can be measured according to ASTM D611 – 07.

[0084] The solvent can also exhibit a relatively high flash point. In one embodiment, the solvent has a flash point of at least 150°C, alternatively at least 170°C or at least 190°C. Flash point can be determined according to ASTM D93 – 10a.

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[0085] The solvent can also have a low evaporation loss as determined by the Noack Method, ASTM D5800 – 10. For example, the solvent can have an evaporation loss of not greater than 15 percent, alternatively not greater than 12 percent or not greater than 10 percent.

[0086] In another embodiment of the invention, the solvent is a low viscosity ester composition. Examples of low viscosity esters include, but are not limited to polyol esters (reaction products of at least one carboxylic acid, i.e., mono-basic or multi-basic carboxylic acid, and at least one polyol) and can include low viscosity complex alcohol esters (reaction products of at least one polyol, multi-basic carboxylic acid and mono-alcohol). Specific examples of polyol esters include, but are not limited to, di-iso tridecyl adipate, diisooctyl ester and trimethylolpropane esters of C₈-C₁₀ acids. A specific example of a carboxylic acid includes, but is not limited to, hexanedioic acid.

[0087] Additional examples of low viscosity esters include esters of monocarboxylic or dicarboxylic acids or their anhydrides (e.g., phthalic acid, succinic acid, alkyl succinic acids and alkenyl succinic acids, maleic acid, azelaic acid, suberic acid, sebacic acid, fumaric acid, adipic acid, linoleic acid dimer, malonic acid, alkylmalonic acids, alkenyl malonic acids) with any one or more of a variety of mono-alcohols (e.g., butyl alcohol, hexyl alcohol, dodecyl alcohol, 2-ethylhexyl alcohol, ethylene glycol, diethylene glycol monoether, propylene glycol). These esters include dibutyl adipate, di(2-ethylhexyl) sebacate, di-n-hexyl fumarate, dioctyl sebacate, diisooctyl azelate, diisodecyl azelate, dioctyl phthalate, didecyl phthalate and dieicosyl sebacate. Other examples of esters include those made from C₅ to C₁₂ monocarboxylic acids and polyols such as neopentyl glycol, pentaerythritol, dipentaerythritol and tripentaerythritol.

Process for Producing the Two Phase Lubricant Composition

[0088] A method for producing the lubricant composition includes a step of mixing together the high viscosity ester, high viscosity ether or combination thereof, and solvent to produce a solution. This step can be carried out at any pressure and temperature suitable to ensure adequate mixing of the components. For example, the solution can be prepared by mixing together the high viscosity ester and/or high viscosity ether and solvent at a temperature of from 5°C to 100°C, preferably from 10°C to 90°C, more preferably from 15°C to 80°C, and a pressure of from 10 psig to 100 psig, more preferably from 14 psig to 50 psig.

[0089] The solution should contain sufficient high viscosity material to affect film height or thickness characteristics of the ultimate lubricant composition. The solvent should be mixed with the high viscosity ester, high viscosity ether or combination thereof to produce a solution containing at least 0.1 wt %, alternatively at least 0.5 wt % or at least 1 wt % solvent. The solution should contain not greater than 10 wt %, alternatively not greater than 8 wt % or not greater than 5 wt % solvent. For example, the solution can contain from 0.1 wt % to 10 wt % solvent, based on total weight of the solution prior to mixing with the low viscosity Group IV base oil.

[0090] After the solution is produced, it is added to the low viscosity Group IV base oil to produce a discontinuous phase dispersed throughout the continuous phase of the lubricating composition. The solution is added in any appropriate manner such as by dripping onto or injecting into the low viscosity Group IV base oil.

[0091] The solution is added to the low viscosity Group IV base oil through appropriate mixing and at a rate effective to produce the desired discontinuous phase. As an example, the solution is added to the low viscosity Group IV base

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oil at a rate of at least 0.1 ml/min, alternatively at least 2 ml/min, or at least 5 ml/min, or at least 10 ml/min, or at least 20 ml/min.

[0092] The solution can be added to the low viscosity Group IV base oil at any pressure and temperature suitable to produce the desired discontinuous phase dispersed throughout the continuous phase of the lubricating composition. For example, the solution can be added to the low viscosity Group IV base oil at a temperature of from 5°C to 100°C, preferably from 10°C to 90°C, more preferably from 15°C to 80°C, and a pressure of from 10 psig to 100 psig, more preferably from 14 psig to 50 psig.

[0093] As another example, the solution can be added to the low viscosity Group IV base oil by heating the low viscosity Group IV base oil and then adding the solution where they dissolve with agitation followed by cooling the mixture. Heating can be carried out by heating the low viscosity Group IV base oil at a temperature of from 20°C to 100°C, preferably from 30°C to 80°C, until the materials are substantially dissolved. The dissolved fluids can then be cooled to a temperature at which the fluids separate into a continuous phase and a discontinuous phase. Cooling will involve a reduction in temperature from the heating temperature of at least 20°C, preferably at least 30°C, and preferably not below 0°C or below 10°C. The formation of a continuous phase and a discontinuous phase at the cooling temperature can be referred to as an emulsion as well as a dispersion.

[0094] The method of this invention is carried out so as to add into the low viscosity Group IV base oil an amount of the high viscosity ester and/or high viscosity ether as a dispersed discontinuous phase component of the lubricant composition sufficient to promote improved lubrication performance relative to the continuous phase base oil alone. The discontinuous phase will also be present in the lubricant in an amount sufficient to promote the formation of a two-phase

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lubricant. As such, an amount of the discontinuous phase component can be present such that it surpasses the critical miscibility concentration in the continuous phase base oil. Generally, the solution of the high viscosity ester and/or high viscosity ether and solvent is added to the low viscosity Group IV base oil to produce a discontinuous phase present in the lubricant in an amount of from about 0.1% to about 10% by weight, or more preferably from about 0.1% to about 5% by weight, or even more preferably from about 0.1% to about 3% by weight, based on total weight of the lubricant produced.

[0095] Microfluidization steps can be incorporated into the process for making the lubricant composition. Microfluidization can be carried out by any suitable microfluidizer for producing either emulsions or dispersions, and that is capable of reducing mean particle size of the discontinuous phase material. The primary forces for microfluidization are:

- a. shear, involving boundary layers, turbulent flow, acceleration and change in flow direction;
- b. impact, involving collision of the particles processed with solid elements of the microfluidizer, and collision between the particles being processed; and
- c. cavitation, involving an increased change in velocity with a decreased change in pressure, and turbulent flow. An additional force can be attributed to attrition, i.e., grinding by friction.

[0096] M-110 series laboratory scale microfluidizers available from Microfluidics™, Newton, MA, can be used according to this invention. These microfluidizers contain an air motor connected to a hydraulic pump, which circulates the process fluid, i.e., the cooled emulsion or dispersion. The fluid is propelled at high pressures (up to 23,000 psi) through a chamber that has fixed

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microchannels for focusing the fluid stream and accelerating it to a high velocity. Within the chamber the fluid is subjected to intense shear, impact and cavitation, all of which contribute to particle size reduction. After processing, the fluid stream is passed through a heat exchanger coil, and the fluid can be collected or recirculated through the machine. The heat exchanger and chamber can be externally cooled with a refrigerated circulating water bath.

[0097] The use of the solvent according to the method of this invention can also be referred to as solvent displacement. A “displacing solvent” can be defined as any solvent or mixture of solvents that has the desired level of miscibility with the discontinuous phase ester composition so that the discontinuous phase ester is replaced with the solvent when the discontinuous phase composition is contacted with continuous phase base oil. In this invention, the displacing solvent is the solvent as described above.

Lubricating Composition Characteristics

[0098] The lubricant composition of this invention has superior lubricating performance. This performance property can be observed in a point contact optical EHL film thickness measurement device in which EHL film thickness is measured as a function of temperature and dynamic viscosity (product of kinematic viscosity and density). EHL film thickness can be expressed as LP, the lubricant parameter, which is a product of the dynamic viscosity, η_0 (cP), and the pressure-viscosity coefficient, α (psi⁻¹), according to equation 1:

$$LP = 10^{11} \eta_0 \alpha \quad (\text{Eq. 1})$$

[0099] As apparent from equation 1, film thickness is expected to increase upon increasing the values for dynamic viscosity or pressure-viscosity coefficient. LP is the lubricant contribution to film thickness in EHL contacts. The lubricant

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parameter (LP) concept is fully described in the industry publication *Mobil EHL Guidebook*, Fourth edition, Mobil Oil Corp., Technical Publications, Fairfax, Va., 1992, herein incorporated by reference.

[0100] The lubricant compositions of this invention can be considered a two phase lubricant, as it is comprised of a continuous phase and a discontinuous phase. It can be used as high performance automotive engine oils, general industrial lubricants, grease, various types of automotive or industrial gears oils, aviation lubricants, hydraulic fluids or lubricants, heat transfer fluids, insulating fluids.

[0101] The two phase lubricating composition preferably has a standard ISO grade of 15 to 3,200 and is used in industrial applications, such as industrial worm gears. It is particularly suited for standard ISO grades 15, 22, 32, 46, 68, 100, 150, 220, 320 and 460. However, in another embodiment, the lubricating composition has a corresponding SAE grade of SAE 75W-90, SAE 80W-90, or SAE 85W-90 to SAE 85W-250, and is used in automotive applications, such as automotive gears.

[0102] In one embodiment, the two phase lubricating composition has a kinematic viscosity of 20 cSt to 3,300 cSt at 40°C and a corresponding ISO VG grade of 15 to 3,200. The lubricating compositions having the ISO VG grades of 15 to 3,200 are acceptable for use in industrial gear applications, such as steel on steel gears or steel on bronze gears.

[0103] In one embodiment, the two phase lubricating composition has a kinematic viscosity of from 10 cSt at 100°C to 200 cSt at 100°C. In another embodiment, the lubricating composition has a kinematic viscosity of from 20 cSt at 100°C to 100 cSt at 100°C. In yet another embodiment, the lubricating composition has a kinematic viscosity of from 50 cSt at 100°C to 100 cSt at

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100°C. The kinematic viscosity is measured according to the ASTM D445 standard test method.

[0104] In one embodiment, the two phase lubricating composition has a viscosity index (VI) of 80 to 300. In another embodiment, the lubricating composition has a viscosity index of 100 to 275. In yet another embodiment, the lubricating composition has a viscosity index of 120 to 250. The viscosity index is measured according to the ASTM D2270 standard test method.

[0105] The blended lubricating composition allows power to be efficiently transported through the machinery in which the lubricating composition is used, so that little power is wasted to friction or heat. The shear stability, viscosity, and other properties of the blended lubricating composition allows the machinery to employ lower operating temperatures, which leads to lower energy consumption and lower energy costs. The lower operating temperature also leads to less degradation of the machinery and seals due to heat, and thus provides a longer machine life and longer seal life.

Examples

Example 1

[0106] Four solvent displacement emulsions were made with kerosine diluent by the following procedure. Esterex™ A51 derived Complex Ester was used with a viscosity of 186 cSt at 100°C at room temperature. A stock solution of 10 wt% of the Esterex™ A51 derived Complex Ester was prepared in Jet A Kerosene. The viscosity of the kerosene was 1 cSt at 40°C. 100g of ExxonMobil Chemical Company SpectraSyn™ 6 (PAO-6) was weighed into a 400 ml beaker. A 2.6 inch magnetic stirrer was placed on the bottom of the beaker. The solution was stirred at a setting of 5.5 (approximately 250 RPM) with a Corning Stir/Hotplate. 0.6 ml

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(0.5 grams) of the EsterexTM solution in kerosine was pipetted dropwise into the stirred PAO-6 at a point halfway between the center of the vortex and the edge of the beaker. The addition rate of high viscosity ester solution varied between 0.125 and 7.2 ml/min. The temperature of the PAO-6 was either 22 or 50°C. The median droplet size of the resulting emulsions was measured with a Horiba LA910 Light Scattering Particle Size Analyzer. The results are presented in Table 1 below. Median drop size ranged between 2.4 and 3.0 microns. Addition rate in the selected range had a minor affect on drop size. Elevated temperature decreased the drop size slightly. Overall, the solvent displacement procedure was able to make micron sized droplets with much less shear than used in microfluidized emulsions.

Table 1
Solvent Displacement Emulsions of 500 ppm EsterexTM A51
Derived Complex Ester in SpectraSynTM 6

Esterex TM Solution Addition Rate, ml/min	PAO-6 Temperature, °C	Median Droplet Size, microns
Slow - 0.125 (one drop every 10 sec)	22	3.0
Rapid - 3.6 (0.6 ml in 10 sec)	22	2.9
Slow - 0.25 (one drop every 5 sec)	50	2.6
Rapid - 7.2 (0.6 ml in 5 sec)	50	2.4

Example 2

[0107] Two solvent displacement emulsions containing 500 ppm Esterex™ A51 derived Complex Ester in SpectraSyn™ 6 (PAO-6) were made by a sub-surface injection method with kerosene diluent and with a low viscosity adipate ester diluent at room temperature (22°C). 10 wt% stock solutions of the Esterex™ A51 derived Complex Ester were prepared in each diluent. The low viscosity adipate ester diluent, Esterex™ A51 was obtained from ExxonMobil Chemical Company. Its viscosity was 5.2 cSt at 100°C. A 250 ml beaker was filled with 141 grams of PAO-6. The emulsions were made by injection of 0.855 and 0.773 ml, respectively, of diluent solution through a 2 inch 26 Gauge needle positioned 1 mm from the surface of a rotating cylindrical steel Premier Dispersator head rotating at 700 RPM beneath the surface of the PAO-6. The addition rate of the diluent solution was 0.1 ml/min controlled by a syringe pump. The resulting median drop size of the emulsion prepared with the kerosene diluent was 1.8 microns and with the low viscosity adipate ester diluent was 0.9 microns. Sub-surface injection in a high shear zone resulted in smaller drop size (1.7 microns) in the emulsion prepared with kerosene diluent at 22°C and a similar addition rate in Example 1 (3.0 microns). The low viscosity adipate ester diluent produced an substantially smaller drop size (0.9 microns). Its viscosity (5.2 cSt at 100°C) was similar to that of the PAO-6 basestock (5.8 cSt at 100°C).

[0108] The film thickness of each of the emulsions was measured in a PCS Instruments EHL Ultra Thin Film Measurement System at 80°C and 20 Newtons of load. The measurement was made with a rotating glass disc and (driven) steel ball at speeds from 1.3 to 0.018 m/s and 0% slide/roll ratio. The results are compared to those for a microfluidized emulsion containing the same concentration of Esterex™ A51 derived Complex Ester in PAO-6 in Figure 1. EHL film thickness of the solvent displacement emulsion prepared with kerosene

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diluent is significantly lower than that of the microfluidized emulsion, while there appears to be no reduction in film thickness in the solvent displacement emulsion prepared with low viscosity adipate ester diluent. It is concluded that the ester diluent produces a substantially greater film thickness relative to the kerosene diluent.

Example 3

[0109] Two solvent displacement emulsions were made by a drop wise process similar to that in Example 1. The emulsions contained 5000 ppm (0.5 wt%) Esterex™ A51 derived Complex Ester (HV Ester) and 0.45 wt% Chemtura Durad 90 antiwear agent in SpectraSyn™ 6 (PAO-6). The D90 antiwear agent and Esterex™ A51 derived Complex Ester were premixed in low viscosity adipate ester (LVEster) diluent at two different concentrations. The first contained 20% Esterex™ A51 derived Complex Ester and 18% D90 in the LVEster. The second contained 10% Esterex™ A51 derived Complex Ester and 9% D90 in the LVEster. The median drop size of the resulting emulsions were 6 and 2 microns respectively.

[0110] The wear performance of the emulsions in Examples 1-3 was measured in a High Frequency Reciprocating Rig at 60 Hertz, 400 grams load and 80°C for 2 hours and compared to a control containing D90, low viscosity adipate ester diluent and PAO-6 and to a microfluidized emulsion containing no diluent and in which the D90 and HVEster had not been premixed. Dramatic reductions in HFRR wear volumes were seen with both solvent displacement emulsions. The large decrease is attributed to forced partitioning of D90 into HVE ester by premixing in the diluent. An exceptionally high concentration of D90 antiwear agent is brought into contact with the metal surface when the polar HV ester droplets adsorb on the surface.

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Example 4. EHL Film Thickness of 0.5 wt% PTE in SpectraSyn™ 8, a Polyalpha Olefin (PAO-8) (Standard Blending)

[0111] A sample of a viscous poly-tetrahydrofuran ester (PTE) was used with a viscosity of 652 cSt at 100°C and density 0.97g/cc at room temperature. SpectraSyn™ 8, a polyalpha olefin having a kinematic viscosity of 8 at 100°C (PAO-8), base oil was obtained from ExxonMobil Chemical Company. A 0.5 wt% dispersion of PTE in PAO-8 was prepared by weighing 1.5 g of PTE and 298.5 g of PAO-8 into a 600ml glass beaker. A 2 inch magnetic stirbar was placed in the beaker and the sample was mixed at a setting of 3.5 on a Corning Model PC/620 Stirrer/Hot Plate while it was heated to 65°C and held at 65°C for 30 minutes. The heat and stirring were turned off and the sample was allowed to cool to room temperature. The median droplet size of the resulting emulsion was measured in a Horiba LA910 Light Scattering Particle Size Analyzer and determined to be 29.5 microns.

[0112] The film thickness of pure PTE, pure PAO-8 and the 0.5 wt% dispersion of PTE in PAO-8 were measured in a PCS Instruments EHL Ultra Thin Film Measurement System at 80°C and 20 Newtons of load. The measurement was made with a rotating glass disc and (driven) steel ball at speeds from 1.3 to 0.018 m/s and 0% slide/roll ratio. The results are shown in Figure 2. The film thickness of the dispersion at the lower speeds is greater than that of pure based PAO-8 implying concentration of PTE within the contact region between the steel ball and the glass disc. The percent of PTE in the contact can be estimated from the following equation:

$$\text{estimated \% dispersed phase in contact} = \frac{h_{\text{DISP}} - h_{\text{PAO}}}{h_{\text{PTE}} - h_{\text{PAO}}} \quad (100)$$

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where h_{DISP} , h_{PAO} and h_{PTE} are EHL film thickness of the dispersion, pure PAO-8 and pure PTE, respectively. By this equation the estimated percent of PTE in the contact is 18% at 0.02 m/s and 5.6% at 0.1 m/s.

Example 5. EHL Film Thickness of 0.5 wt% PTE in SpectraSyn™ 8, a Polyalpha Olefin (PAO-8) (Microfluidized)

[0113] A emulsion of PTE dispersed in PAO-8 was prepared according to the standard blending procedure in Example 4. The median droplet size of the emulsion was determined to be 35.4 microns with the Horiba Particle Size Analyzer. The emulsion was then passed through a lab scale model 110T microfluidizer 4 times at a pressure of 14,000 psi. The median particle size of the emulsion was reduced to 2.1 microns. The EHL film thickness of the microfluidized emulsion was measured under the same conditions as in Example 4 and the results are plotted in Figure 2. The film thickness of the microfluidized emulsion is significantly higher than that of the standard blended emulsion at all speeds. The estimated percent of PTE in the contact at 0.02 m/s is 27% and at 0.1 m/s is 23% in comparison to 18% and 5.6% for the standard blended emulsion.

Example 6. EHL Film Thickness of 0.5 wt% PTE in PAO-8 (SpectraSyn™ 8) (Standard Blending)

[0114] PAO-8 was obtained from ExxonMobil Chemical Company. An emulsion of PTE dispersed in PAO-8 was prepared according to the standard blending procedure in Example 4. The median droplet size of the emulsion was determined to be 44.8 microns. The EHL film thickness of the emulsion was measured under the same conditions as in Example 4 and the results are plotted in Figure 3. The film thickness of the standard blended emulsion in PAO-8 is lower than that of the standard blended emulsion in PAO-8 especially in the speed range of 0.02 to 0.1 m/s. The estimated percent of PTE in the contact at 0.02 m/s is

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8.3% and at 0.1 m/s is 4.0% as compared to 18 % and 5.6% for the standard blended emulsion in PAO-8. PAO-8 appears to reduce the EHL effect of the standard blended PTE emulsion.

Example 7. EHL Film Thickness of 0.5 wt% PTE in PAO-8 (SpectraSyn™ 8) (Microfluidized)

[0115] An emulsion of PTE dispersed in PAO-8 was prepared according to the standard blending procedure in Example 4. The median droplet size of the emulsion was determined to be 31.3 microns. The emulsion was then passed through a lab scale model 110T microfluidizer 4 times at a pressure of 14,000 psi. The median droplet size of the emulsion was reduced to 1.6 microns. The EHL film thickness of the microfluidized emulsion was measured under the same conditions as in Example 4 and the results are plotted in Figure 3. The film thickness of the microfluidized emulsion is again significantly higher than that of the standard blended emulsion. The estimated percent of PTE in the contact at 0.02 m/s is 18% and at 0.1 m/s is 18 % in comparison to 8.3 % and 4.0 % for the standard blended emulsion. Microfluidization minimizes the solvent effect. This will be an advantage in commercial lubricant formulations.

Example 8. EHL Film Thickness of 0.5 wt% Esterex™ A51 Derived Complex Ester in PAO-8 (SpectraSyn™ 8) (Standard Blending)

[0116] Esterex™ A51 derived Complex Ester was used with a viscosity of 186 cSt at 100°C and density was 1.05 g/cc at room temperature. A 0.5 wt% dispersion of the Ester in PAO-8 was prepared by the procedure described in Example 4. The median droplet size of the emulsion was 55.0 microns. Its film thickness was measured under the conditions described in Example 4 and is shown in Figure 4. The estimated percent of Ester in the contact is 39% at 0.02

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m/s and 26% at 0.1 m/s in comparison to 18% and 5.6% for standard blended PTE in Example 1.

Example 9. EHL Film Thickness of 0.5 wt% EsterexTM A51 Derived Complex Ester in PAO-8 (SpectraSynTM 8) (Microfluidized)

[0117] A 400g dispersion of 0.5 wt% EsterexTM A51 derived Complex Ester in PAO-8 was prepared by the method described in Example 4. The median particle size of the normally blended emulsion was 63.3 microns. The dispersion was passed through the microfluidizer 4 times at 14,000 psi and the median particle size was reduced to 3.5 microns. The film thickness of the microfluidized emulsion was measured under the conditions described in Example 4 and the results are shown in Figure 4. The film thickness of the dispersion is close to that of pure Ester in the speed range of 0.02 to 0.1 m/s indicating a highly flooded contact in that speed range. The estimated percent of Ester in the contact is 74% at 0.02 m/s and 55% at 0.1 m/s.

Example 10. EHL Film Thickness of 0.5 wt% EsterexTM A51 Derived Complex Ester in PAO-8 (SpectraSynTM 8) (Standard Blending)

[0118] An emulsion of EsterexTM A51 derived Complex Ester dispersed in PAO-8 was prepared according to the standard blending procedure in Example 4. The median droplet size of the emulsion was determined to be 54.6 microns. The EHL film thickness of the emulsion was measured under the same conditions as in Example 4 and the results are plotted in Figure 5. The estimated percent of EsterexTM A51 derived Complex Ester in the contact at 0.02 m/s is 25% and at 0.1 m/s is 4.0%, much lower than that for the standard blended emulsion of EsterexTM A51 derived Complex Ester in PAO-8. PAO-8 appears to reduce the EHL effect of the standard blended EsterexTM Complex Ester emulsion as it did for PTE emulsions.

Example 11. EHL Film Thickness of 0.5 wt% Esterex™ A51 Derived Complex Ester in PAO-8 (SpectraSyn™ 8) (Microfluidized)

[0119] An emulsion of Esterex™ A51 derived Complex Ester dispersed in PAO-8 was prepared according to the standard blending procedure in Example 4. The median droplet size of the emulsion was determined to be 54.1 microns. The emulsion was then passed through a lab scale model 110T microfluidizer 4 times at a pressure of 14,000 psi. The median particle size of the emulsion was reduced to 1.7 microns. The EHL film thickness of the microfluidized emulsion was measured under the same conditions as in Example 4 and the results are plotted in Figure 5. Again, the film thickness of the microfluidized emulsion is significantly higher than that of the standard blended emulsion. The estimated percent of Esterex™ A51 derived Complex Ester in the contact at 0.02 m/s is 76% and at 0.1 m/s is 55% in comparison to 25% and 4.0% for the standard blended emulsion. Microfluidization again gives an improvement versus normal blending in EHL performance.

Example 12. MTM Friction Coefficient of 0.5 wt% Esterex™ A51 Derived Complex Ester in PAO-8 (SpectraSyn™ 8) (Microfluidized)

[0120] Stribeck curves were generated for pure PAO-8, pure Esterex™ A51 derived Complex Ester and the microfluidized 0.5 wt% dispersion of Ester in PAO-8 in Example 11 using a PCS Instruments Mini Traction Machine. The conditions of the experiment were 80°C, 0.5 GPa pressure and 30% slide to roll ratio at speeds of 3 to 0.002 m/s. The results are shown in Figure 6. The friction coefficient of pure PAO-8 increased as speed was reduced below 0.1 m/s. As film thickness decreases, contact between the steel surfaces causes friction to increase. By contrast, the friction coefficient remained low at all speeds with pure Complex Ester. The friction coefficient of the emulsion tracked pure PAO-8 and Complex Ester from 3 to 0.15 m/s, rose slightly above PAO-8 between 0.15 and 0.035 m/s

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and then dropped significantly below PAO-8 at speeds lower than 0.035 m/s suggesting concentration of the Ester in the contact region as in the EHL experiment. These results demonstrate that enhanced film thickness persists in the presence of shear (30% slide/roll ratio) and in steel-on-steel contacts.

[0121] The principles and modes of operation of this invention have been described above with reference to various exemplary and preferred embodiments. As understood by those of skill in the art, the overall invention, as defined by the claims, encompasses other preferred embodiments not specifically enumerated herein.

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CLAIMS:

1. A method for producing a lubricant composition having a discontinuous phase dispersed throughout a continuous phase, the method comprising the steps of:

mixing together high viscosity ester, high viscosity ether or combination thereof, and polar solvent to produce a solution, wherein the high viscosity ester, high viscosity ether or combination of high viscosity ester and ether have a viscosity of at least 100 cSt at 100°C; and

adding at least a portion of the solution to a low viscosity Group IV base oil having a viscosity of less than 100 cSt at 100°C to produce the discontinuous phase dispersed throughout the continuous phase,

wherein the discontinuous phase is comprised of droplets of the high viscosity ester having a mean average diameter of from 0.1 microns to 20 microns.

2. The method of claim 1, wherein the solvent has a viscosity of from 1 cSt to 100 cSt at 100°C.

3. The method of claim 1, wherein the solvent has a viscosity of from 1 cSt to 80 cSt at 100°C.

4. The method of claim 3, wherein the high viscosity ester, high viscosity ether and the low viscosity Group IV base oil are miscible in the solvent.

5. The method of claim 1, wherein the solvent is an aprotic polar composition.

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6. The method of claim 1, wherein the solvent has an aniline point of not greater than 45°C.
7. The method of claim 1, wherein the solvent has a flash point of at least 150°C.
8. The method of claim 1, wherein the solvent is a low viscosity ester composition.
9. The method of claim 7, wherein the low viscosity ester solvent is comprised of adipate ester.
10. The method of claim 1, wherein the high viscosity ester and high viscosity ether are more highly miscible in the solvent than the low viscosity Group IV base oil.
11. The method of claim 1, wherein at least a portion of the high viscosity ester composition is comprised of an ester containing at least one ether linkage.
12. The method of claim 1, wherein at least a portion of the high viscosity ester composition is comprised of an ester containing no ether linkages.
13. The method of claim 1, wherein the solution contains from 0.1 wt % to 10 wt % solvent, based on total weight of the solution prior to mixing with the low viscosity Group IV base oil.
14. The low viscosity Group IV base oil has, independently, a viscosity of from 1 cSt to 100 cSt at 100°C.

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15. The method of claim 1, wherein the solution is added to the low viscosity Group IV base oil at a rate of at least 0.1 ml/min.

16. The method of claim 1, wherein the solution is added to the low viscosity Group IV base oil at a temperature of from 20°C to 80°C.

17. The method of claim 1, wherein the lubricant composition is microfluidized following production of the dispersed phase.

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Figure 1. EHL Results for Solvent Displacement Emulsions Compared to a Microfluidized Emulsion

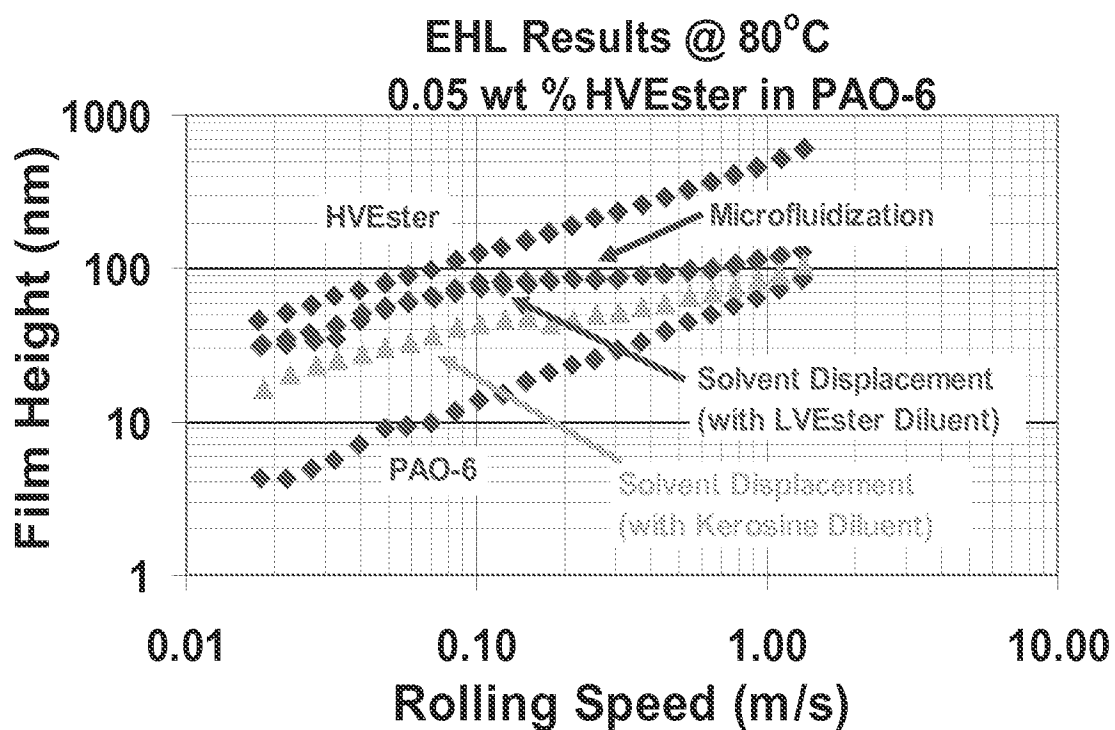


Figure 1

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Figure 2. EHL Film Thickness of 0.5 wt% Dispersions of PTE in PAO-8 in Comparison to Pure PAO-8 and Pure PTE

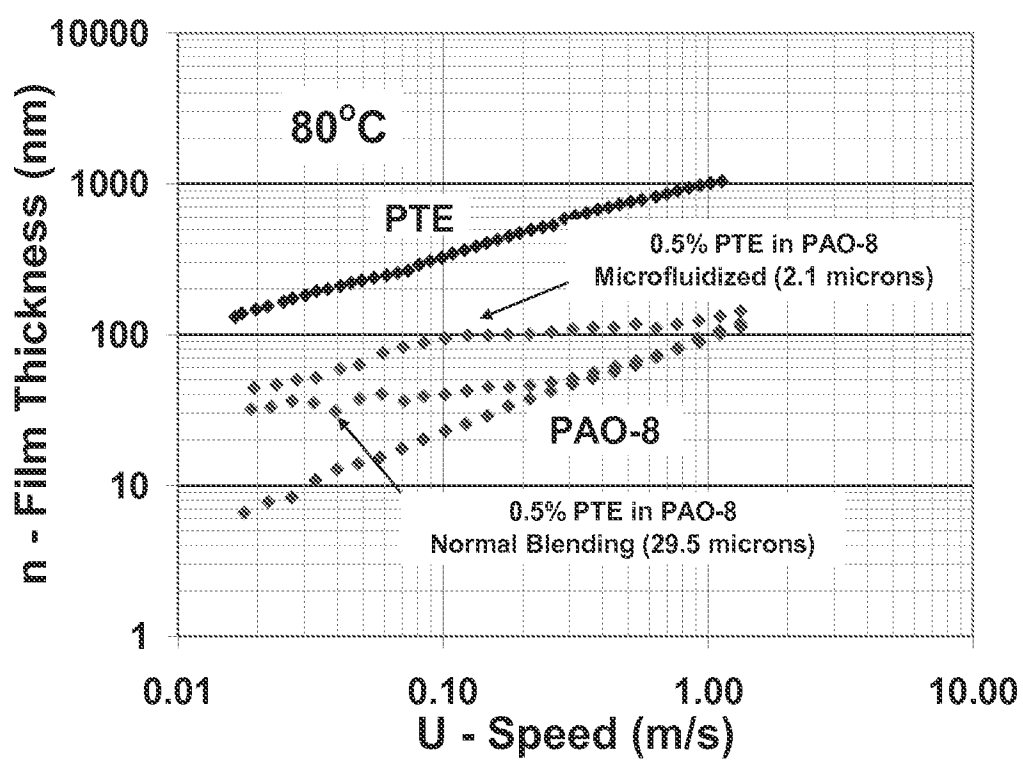


Figure 2

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Figure 3. EHL Film Thickness of 0.5 wt% Dispersions of PTE in PAO-8 in Comparison to Pure PAO-8 and Pure PTE

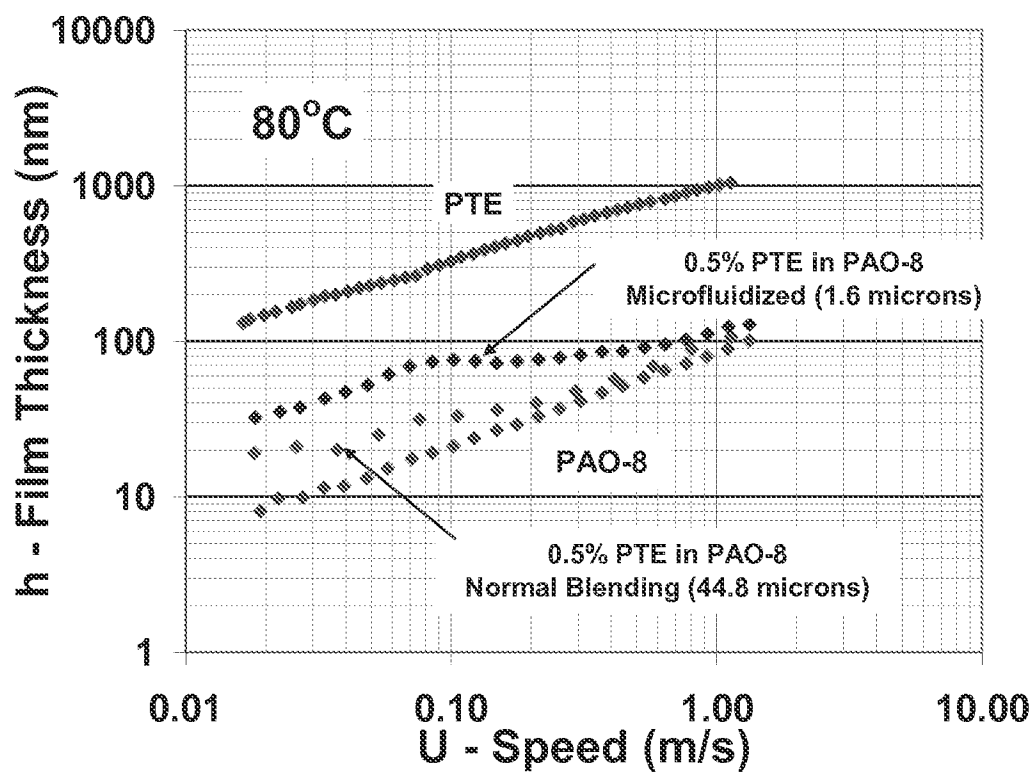


Figure 3

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Figure 4. EHL Film Thickness of 0.5% Complex Ester Dispersions in PAO-8 in Comparison to Pure PAO-8 and Pure Complex Ester

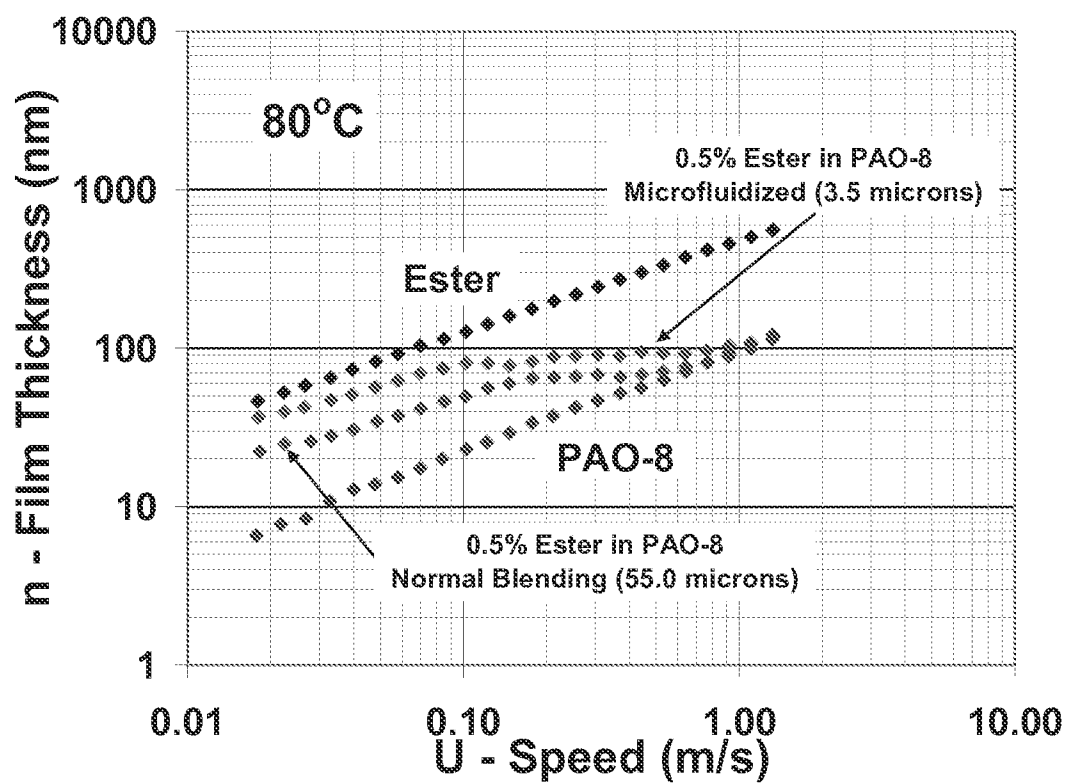


Figure 4

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Figure 5. EHL Film Thickness of 0.5% Complex Ester Dispersions in PAO-8 in Comparison to Pure PAO-8 and Pure Complex Ester

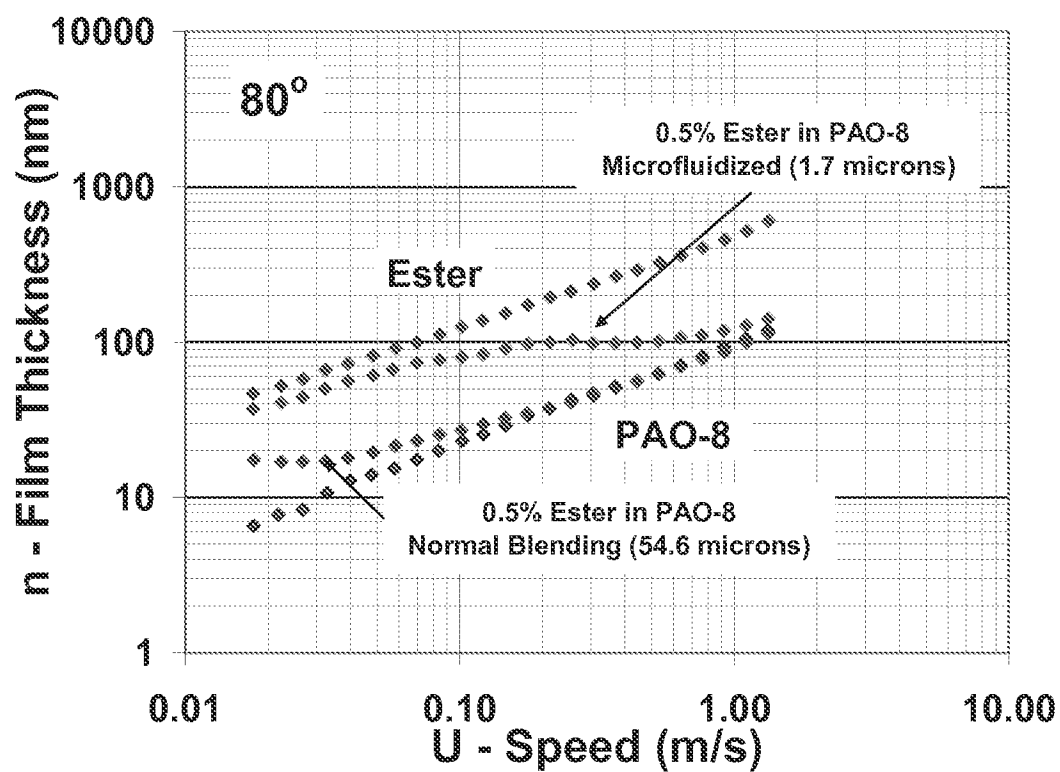


Figure 5

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Figure 6. Friction Coefficient of Microfluidized 0.5% Complex Ester in PAO-8 Compared to Pure PAO-8 and Pure Complex Ester

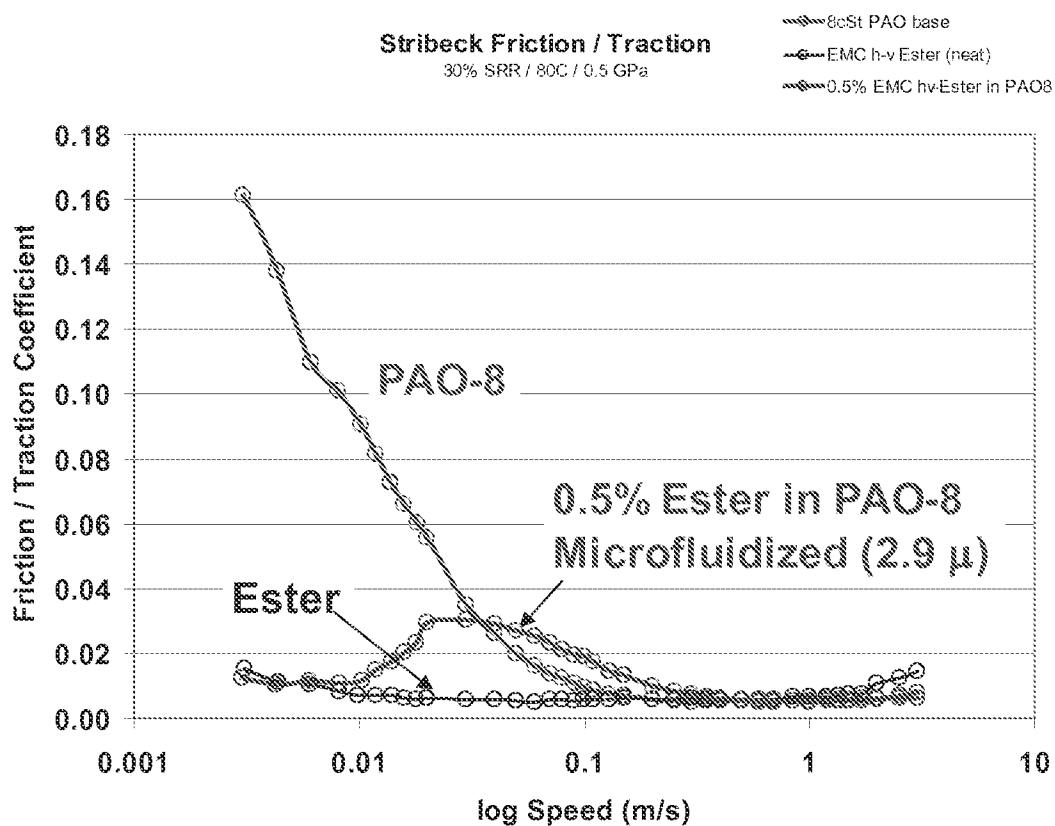


Figure 6

INTERNATIONAL SEARCH REPORT

International application No

PCT/US2012/039507

A. CLASSIFICATION OF SUBJECT MATTER

INV. C10M111/00
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C10M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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A	US 2007/189998 A1 (NAIR MRIDULA [US] ET AL) 16 August 2007 (2007-08-16) the whole document -----	1-17



Further documents are listed in the continuation of Box C.



See patent family annex.

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Date of the actual completion of the international search

24 July 2012

Date of mailing of the international search report

31/07/2012

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

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