



(11) **EP 2 292 724 A1**

(12) **EUROPEAN PATENT APPLICATION**

(43) Date of publication:
09.03.2011 Bulletin 2011/10

(51) Int Cl.:
C10M 171/02 (2006.01) **C10M 169/04** (2006.01)
C10M 167/00 (2006.01) **C10M 163/00** (2006.01)

(21) Application number: **10179036.8**

(22) Date of filing: **22.07.2005**

(84) Designated Contracting States:
FR GB

(30) Priority: **29.07.2004 US 592356 P**
11.03.2005 US 660650 P

(62) Document number(s) of the earlier application(s) in
accordance with Art. 76 EPC:
05775264.4 / 1 778 824

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Remarks:

This application was filed on 23-09-2010 as a
divisional application to the application mentioned
under INID code 62.

(54) **Lubricating compositions**

(57) A method of lubricating an internal combustion engine with a power output of at least 1600 kilowatts, with a lubricating composition, the method comprising:
(1) monitoring one or more performance characteristics of the engine; (2) selecting an additive package to provide a desired TBN level to a lubricating composition to modify the performance characteristics of the engine; (3) combining the additive package of step (2) with (a) a compo-

nent having a viscosity of about 2 mm²/s to 12 mm²/s, comprising a light neutral base oil, and optionally (b) a component having a viscosity of above 12 mm²/s to about 40 mm²/s, comprising heavy neutral base oil or a bright-stock, to form a lubricating composition; and (4) supplying the lubricating composition of step (3) to the engine.

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Description

[0001] This application claims priority from US 60/592,356, 20 Jul 2004 and US 60/660,650, 11 Mar 2005.

5 FIELD OF INVENTION

[0002] The present invention relates to a method of lubricating an internal combustion engine by monitoring engine performance and adding an additive package accordingly. The invention further relates to a composition suitable for the method.

10 BACKGROUND OF THE INVENTION

[0003] It is known to add various additives to an oil of lubricating viscosity for diesel or gasoline engines, for instance, a 2-stroke engine, to reduce wear and improve cleanliness. In diesel engines an oil of lubricating viscosity is used particularly to reduce wear of cylinder liners and piston rings. Often engine operating temperatures and pressures are sufficient to break down the film of the oil of lubricating viscosity on the internal walls of the cylinder, and increased wear and decreased engine cleanliness due to deposits occurs.

[0004] In many 2-stroke engines, especially marine diesel engines or stationary power diesel engines, there are two lubricating compositions. One composition is a system oil of viscosity generally less than 12 mm²/s which is used to lubricate the crankcase of the 2-stroke engines and has a low Total Base Number (TBN). The system oil is normally unsuitable for lubricating cylinder liners and piston rings because of its low TBN and low viscosity. The second lubricating composition used in a 2-stroke engine has a higher viscosity and TBN and is suitable for lubricating cylinder liners and piston rings. This is sometimes referred to as a cylinder oil. (All viscosities reported herein are kinematic viscosity measured at 100°C, unless otherwise specified).

[0005] Further, in 2-stroke engines associated with marine vessels or stationary power stations, typical fully formulated lubricating compositions suitable for lubricating cylinder liners and piston rings have a total base number of 70 to 80. However, due to variations in sulphur content of fuel, the amount of TBN required varies as amount of base required to neutralise sulphuric acid produced during combustion changes. The presence of excessive amounts of unreacted compounds in an additive package providing TBN, e.g., detergents, can contribute to the formation of deposits. To overcome differences in the sulphur content of fuel, two or more lubricating compositions with differing TBN are independently available for use. One lubricating composition has TBN of 40 to 50 suitable for low sulphur containing fuel, whereas the second lubricating composition has a TBN of 70 or more and is used for higher sulphur containing fuel.

[0006] Increased amounts of deposits and contaminants from cylinder wear are known to contaminate system oil and/or other unused oil suitable for lubricating the cylinder liners and piston rings. When contaminated, the system oil and/or oil suitable for lubricating the cylinder liners and piston rings needs to be replenished to prevent contaminants having a detrimental effect on performance.

[0007] When the system oil is contaminated to levels exceeding those defined in CIMAC guidelines titled "Guidelines for the Lubrication of Two-Stroke Crosshead Diesel Engines," (page 42, section 8, 1997), it is removed from the crankcase and replenished with fresh system oil. The removed system oil is typically stored in a sludge tank until it undergoes waste management treatment.

[0008] US Patent Application 2003/0196632 A1 discloses a method to employ instrumentation to effectuate variation in lubricant flow rate in response to actual engine conditions. The method regularly monitors one or more engine parameters with instruments such as XRF or IR for base number measurement. The measured engine parameters are used to calculate the feed rate of lubricant to the engine.

[0009] US Patent Application 2003/0159672 A1 discloses a method of regularly monitoring one or more engine parameters of an all-loss lubricating system and calculating from the engine parameters an amount of a secondary fluid that is required to be added to base fluid to create a modified base lubricant that is applied to the engine during operations.

[0010] US Patent Application 2003/0183188 A1 discloses a device and a process for real time optimizing engine lubricating oil properties in response to actual operating conditions. The process includes on-line modification of lubricant properties by repeatedly measuring a system that recirculates a base lubricant and one or more system condition parameters at a location of interest. The process then calculates an amount of secondary fluid to add to the lubricant followed by mixing the base fluid with the secondary fluid creating a modified base lubricant and applying to a location of interest.

[0011] International Application WO 99/64543 A1 discloses diesel cylinder oil having a viscosity of 15 to 27 mm²/s (or cSt), a viscosity index of at least 95 and a TBN of at least 40 mg KOH/g. The oil is a neutral base stock of no more than 725 SUS viscosity at 100°C and 2 to 15 wt % of the oil a liquid polyisobutylene with a viscosity of 1500 to 8000 mm²/s (cSt) at 100°C.

[0012] Therefore it would be advantageous to provide a lubricating composition and method of imparting the lubricating

composition with the correct properties to control deposit formation, wear and reduced waste treatment costs. The present invention provides a lubricating composition and a method of imparting a lubricating composition with such properties using a selected additive package to control deposit formation or wear.

SUMMARY OF THE INVENTION

[0013] In one embodiment the present invention provides a method of lubricating an internal combustion engine with a power output of at least 1600 kilowatts, with a lubricating composition, the method comprising:

- (1) monitoring one or more performance characteristics of the engine;
- (2) selecting an additive package to provide a desired TBN level to a lubricating composition to modify the performance characteristics of the engine;
- (3) combining the additive package of step (2) with (a) a component having a viscosity of 2 mm²/s to 12 mm²/s, comprising a light neutral base oil, and optionally (b) a component having a viscosity of above 12 mm²/s to 35 mm²/s or 40 mm²/s, comprising heavy neutral base oil or a brightstock, to form a lubricating composition; and
- (4) supplying the lubricating composition of step (3) to the engine,

wherein the lubricating composition has a total base number of at least 10 mg KOH/g; and the component comprising the light neutral base oil is supplied from an oil reservoir available to said internal combustion engine.

[0014] In one embodiment the invention further provides a method of lubricating an internal combustion engine with a power output of at least 1600 kilowatts, with a lubricating composition, the method comprising contacting (that is, lubricating or supplying) the internal combustion engine with a lubricating composition comprising:

- (a) a 2 mm²/s to 12 mm²/s light neutral base oil;
- (b) 0 to 20 weight percent of a heavy neutral base oil or a brightstock with a viscosity above 12 mm²/s to 40 mm²/s; and
- (c) at least one performance additive comprising: a polymeric thickener, a dispersant, an antiwear agent, a detergent or mixtures thereof; wherein the polymeric thickener, when present, has a weight average molecular weight (Mw) of more than 8000 and is present in an amount to provide a total viscosity of the composition of 12 mm²/s to 29 mm²/s; the dispersant or detergent when present imparts basicity to said lubricating composition; and the composition has a total base number of at least about 10 mg KOH/g.

[0015] In one embodiment the invention further provides a lubricating composition comprising:

- (a) a 2 mm²/s to 12 mm²/s light neutral base oil;
- (b) a heavy neutral base oil or a brightstock with a viscosity of above 12 mm²/s to 35 mm²/s or 40 mm²/s; and
- (c) at least one performance additive comprising: a polymeric thickener, a dispersant, an antiwear agent, a detergent or mixtures thereof;

wherein the polymeric thickener when present has a weight average molecular weight (Mw) of more than 8000 and is present in an amount to provide a total viscosity of the composition of 12 mm²/s or 15 mm²/s to 26.1 mm²/s or 29 mm²/s; the dispersant or detergent when present imparts basicity to said lubricating composition; and the composition has a total base number of at least 10 mg KOH/g.

DETAILED DESCRIPTION OF THE INVENTION

[0016] The present invention provides a method for lubricating an internal combustion engine as described above.

[0017] As used herein all viscosity measurements of the light neutral base oil of heavy neutral base oil are at 100 °C and quoted in units of mm²/s (or cSt).

[0018] In one embodiment the heavy neutral base oil or brightstock has a viscosity of above 12 mm²/s to 35 mm²/s and in another embodiment a viscosity of above 12 mm²/s to 40 mm²/s.

[0019] In one embodiment total viscosity of the lubricating composition is 12 mm²/s or 15 mm²/s to 26.1 mm²/s and in another embodiment 12 mm²/s or 15 mm²/s to 21.9 mm²/s. Typically the lubricating composition with a total viscosity of 12 mm²/s or 15 mm²/s to 26.1 mm²/s is an SAE 60 grade, and an SAE 50 grade lubricating composition has a viscosity of 12 mm²/s or 15 mm²/s to 21.9 mm²/s.

[0020] The performance characteristics of an engine which may be monitored include wear, engine load, variation in TBN, deposits, or corrosion, and these may be monitored directly or indirectly. It is to be understood that the term "monitoring performance characteristics of the engine" not only includes mechanical or power output measurements, but it further includes chemical or physical properties of the lubricating oil in the engine. Wear may be measured by a

number of techniques including determining the metal or metal oxide particles present in scrape down lubricant from a cylinder liner. Other examples of monitoring engine performance include measuring the sulphur content of the fuel, the load of an engine and TBN of the lubricant. A more detailed description of possible techniques for monitoring performance characteristics of an engine is disclosed in US Patent Application 2003/0159672.

[0021] The selection of an additive package to provide a desired TBN level to a lubricating composition may be determined by analyzing fuel properties such as sulphur content or other performance characteristics described above. In one embodiment when the sulphur content of the fuel is low (often less than 4 wt % of the fuel) the amount of TBN required to neutralise acids produced during combustion, e.g., sulphuric acid, is reduced and the additive package may contain less detergent. The TBN provided by the additive package is typically provided in large part by the presence in the package of an overbased detergent, described in greater detail below. In one embodiment the sulphur content of the fuel is high (often over 4 wt % of the fuel) and as a consequence the amount of TBN required from detergent may be higher.

[0022] The total base number (TBN) of the lubricating composition in one embodiment is 30 or higher, in another embodiment 40 or higher, in another embodiment 50 or higher, in another embodiment 60 or higher, in another embodiment 65 or higher and in another embodiment 70 or higher. Examples of the TBN of the lubricating composition include 40, 50, 60, 65, 70, 80 or 100.

[0023] The selected additive package of step (2) is combined with a base oil defined above by known methods such as in a blender. In one embodiment the blender is typically located in situ available relative to an internal combustion engine, which will typically have a power output of at least 1600 kilowatts. In one embodiment the internal combustion engine has a power output of at least 2000 kilowatts, in another embodiment at least 3000 kilowatts and in another embodiment at least 4700 kilowatts.

[0024] The lubricating composition of step (3) in one embodiment is supplied to the combustion engine directly from a "day tank" (or cylinder oil service tank) and in another embodiment from a storage tank.

[0025] In one embodiment the invention provides a lubricating composition comprising:

- (a) a 2 mm²/s to 12 mm²/s light neutral base oil;
- (b) a heavy neutral base oil or a brightstock with a viscosity of above 12 mm²/s to 40 mm²/s; and
- (c) at least one performance additive comprising: a polymeric thickener, a dispersant, an antiwear agent, a detergent or mixtures thereof;

wherein the polymeric thickener when present has a weight average molecular weight (Mw) of more than 8000 and is present in an amount to provide a total viscosity of the composition of 15 mm²/s to 29 mm²/s; the dispersant or detergent, when present, imparts basicity to said lubricating composition; and the composition has a total base number of at least 10 mg KOH/g.

[0026] In one embodiment the invention further provides a lubricating composition comprising:

- (a) a 2 mm²/s to 12 mm²/s light neutral base oil;
- (b) a heavy neutral base oil or a brightstock with a viscosity above 12 mm²/s to 40 mm²/s;
- (c) a polymeric thickener; and
- (d) at least one performance additive comprising: a dispersant, an antiwear agent, a detergent or mixtures thereof;

wherein the polymeric thickener has a weight average molecular weight (Mw) of more than 8000 and is present in an amount to provide a total viscosity of the composition of 15 mm²/s to 29 mm²/s; the dispersant or detergent when present imparts basicity to said lubricating composition; and the composition has a total base number of at least 10 mg KOH/g.

[0027] In one embodiment the invention provides a lubricating composition comprising:

- (a) a 2 mm²/s to 12 mm²/s light neutral base oil;
- (b) 0 to 20 weight percent of a heavy neutral base oil or a brightstock with a viscosity above 12 mm²/s to 40 mm²/s;
- (c) a polymeric thickener; and
- (d) at least one performance additive comprising: a dispersant, an antiwear agent, a detergent or mixtures thereof;

wherein the polymeric thickener has a weight average molecular weight (Mw) of more than 8000 and is present in an amount to provide a total viscosity of the lubricating composition of 15 mm²/s to 29 mm²/s (or viscosity grade ranging from SAE 30 to SAE 60 (typically SAE 50)); the dispersant or detergent when present imparts basicity to said lubricating composition; and the composition has a total base number of at least 10 mg KOH/g.

[0028] In one embodiment the invention employs oil of lubricating viscosity with a 2 mm²/s to 12 mm²/s (typically with an SAE 30 grade) light neutral base oil and optionally a heavy neutral base oil or a brightstock with a viscosity above 12 mm²/s to 40 mm²/s. Such oils include natural and synthetic oils, oil derived from hydrocracking, hydrogenation, and

hydrofinishing, unrefined, refined and re-refined oils and mixtures thereof. Hydrotreated naphthenic oils are also known and can be used, as well as oils prepared by a Fischer-Tropsch gas-to-liquid synthetic procedure as well as other gas-to-liquid oils. In one embodiment the dispersant mixture of the present invention is useful when employed in a gas-to-liquid oil.

[0029] The source of the light neutral base oil in one embodiment is a new or used crankcase system oil from a 2-stroke engine or fresh from a system oil tank without further processing. In one embodiment the used system oil is additised with an additive package to make it useful as a cylinder lubricant. In one embodiment the used system oil is additised with an additive package to prolong oil life or improve performance of the system oil based on the monitoring data of step (1).

[0030] When present, the heavy neutral base oil or brightstock, in one embodiment, is used oil from a sump used to lubricate the internal combustion engine.

[0031] In one embodiment the lubricating composition is an SAE 50 grade lubricant.

[0032] The light neutral base oil is present in one embodiment from 40 to 99.9, in another embodiment 50 to 99.9, in another embodiment 60 to 99.9 and in another embodiment 70 to 99.9 weight percent of the lubricating composition.

[0033] The heavy neutral base oil or brightstock is present in one embodiment from 0 to 35, in another embodiment 0 to 30, in another embodiment 0.01 to 25 and in another embodiment 0.05 to 20 weight percent of the lubricating composition. In one embodiment the heavy neutral base oil is present from 1 to 25 or 5 to 20 weight percent of the lubricating composition.

[0034] In several other embodiments the amount of heavy neutral base oil or brightstock is present from 0 to 20 weight percent, or 0 to 10 weight percent of 0 to 5 weight percent of the lubricating composition. In one embodiment the amount of heavy neutral base oil or brightstock is zero weight percent.

Performance Additives

[0035] Optionally the lubricating composition includes at least one performance additive selected from the group consisting of metal deactivators, polymeric thickeners, dispersants, antioxidants, antiwear agents, corrosion inhibitors, antiscuffing agents, extreme pressure agents, foam inhibitors, demulsifiers, friction modifiers, pour point depressants and mixtures thereof. Typically, fully-formulated lubricating oil will contain one or more of these performance additives.

[0036] The total combined amount of the optional performance additives present in one embodiment from 0 or 0.01 to 25, in another embodiment 0 or 0.01 to 20, in another embodiment 0 or 0.01 to 15 and in another embodiment 0.05 or 0.1 or 0.5 to 10 weight percent of the lubricating composition.

The Polymeric Thickener

[0037] The polymeric thickener includes styrene-butadiene rubbers, ethylenepropylene copolymers, hydrogenated styrene-isoprene polymers, hydrogenated radical isoprene polymers, poly(meth)acrylate acid esters, polyalkyl styrenes, polyolefins (such as polyisobutylene), polyalkylmethacrylates and esters of maleic anhydride-styrene copolymers. In one embodiment the polymeric thickener is free of polyisobutylene; and in another embodiment the polymeric thickener is a polyisobutylene. In one embodiment the polymeric thickener is poly(meth)acrylate.

[0038] The polymeric thickener in several embodiments has a weight average molecular weight (Mw) of more than 8000, or 8400 or more, at least 10,000, or at least 15,000, or at least 25,000 or at least 35,000. The polymeric thickener generally has no upper limit on Mw, however in one embodiment the Mw is less than 2,000,000 in another embodiment less than 500,000 and in another embodiment less than 150,000. Examples of suitable ranges of Mw include in one embodiment 12,000 to 1,000,000, in another embodiment 20,000 to 300,000 and in another embodiment 30,000 to 75,000.

[0039] If a polyisobutylene polymeric thickener is present, the weight average molecular weight (Mw) may be more than 8000, or 8400 or more, at least 10,000 to 15,000 or 25,000. Examples of a suitable range include more than 8000 to 25,000 or 8400 to 15,000.

[0040] The polymeric thickener in one embodiment is present from 0 or 0.01 to 15 and in another embodiment 0.05 to 10 weight percent of the lubricating composition.

Antiwear Agent

[0041] In one embodiment of the invention the lubricating composition further comprises an antiwear agent such as a metal hydrocarbyl dithiophosphate. Examples of a metal hydrocarbyl dithiophosphate include zinc dihydrocarbyl dithiophosphates (often referred to as ZDDP, ZDP or ZDTP). Examples of suitable zinc hydrocarbyl dithiophosphates compounds include the reaction product(s) of butyl/pentyl, heptyl, octyl, and/or nonyl dithiophosphoric acid zinc salts or mixtures thereof.

[0042] In an alternative embodiment the antiwear agent is ashless, i.e., the antiwear agent is metal-free (prior to mixture with other components). In one embodiment the metal-free antiwear agent is an amine salt. The ashless antiwear agent often contains an atom including sulphur, phosphorus, boron or mixtures thereof.

5 Detergent

[0043] The invention optionally includes a detergent such as an overbased sulphonate detergent. The sulphonate detergent of the composition includes compounds represented by the formula:



wherein each R^1 is a hydrocarbyl group in one embodiment containing 6 to 40 carbon atoms, in another embodiment 8 to 35 carbon atoms and in another embodiment 12 to 30 carbon atoms; A may be independently a cyclic or acyclic divalent or multivalent hydrocarbon group; M is hydrogen, a valence of a metal ion, an ammonium ion or mixtures thereof; and k is an integer of 0 to 5, for example 0, 1, 2, 3, 4, 5. In one embodiment k is 1, 2 or 3, in another embodiment 1 or 2 and in another embodiment 1. In one embodiment M is hydrogen and is present on less than 30%, in another embodiment less than 20%, in another embodiment less than 10% and in another embodiment less than 5% of the available M entities, the balance of the M entities being a metal or ammonium ion.

[0044] In one embodiment k is 1 and R^1 is a branched alkyl group with 6 to 40 carbon atoms. In one embodiment k is 1 and R^1 is a linear alkyl group with 6 to 40 carbon atoms.

[0045] Examples of suitable sulphonic acids capable of forming the overbased sulphonate detergent include polypropene benzene sulphonic acid, undecyl benzene sulphonic acid, dodecyl benzene sulphonic acid, tridecyl benzene sulphonic acid, tetradecyl benzene sulphonic acid, pentadecyl benzene sulphonic acid, hexadecyl benzene sulphonic acid and mixtures thereof. In one embodiment the sulphonic acid includes tridecyl benzene sulphonic acid, tetradecyl benzene sulphonic acid, octadecyl benzene sulphonic acid, tetraeicosyl benzene sulphonic acid or mixtures thereof. In one embodiment of the invention the sulphonic acid is a polypropene benzene sulphonic acid, where the polypropene contains 18 to 30 carbon atoms.

[0046] In one embodiment of the invention the sulphonate components are calcium polypropene benzenesulphonate and calcium monoalkyl and dialkyl benzenesulphonates wherein the alkyl groups contain at least 10 or 12 carbons, for example 11, 12, 13, 14, 15, 18, 24 or 30 carbon atoms.

[0047] When M is a valence of a metal ion, the metal may be monovalent, divalent, trivalent or mixtures of such metals. When monovalent, the metal M includes an alkali metal such as lithium, sodium, or potassium, and when divalent, the metal M includes an alkaline earth metal such as magnesium, calcium or barium. In one embodiment the metal is an alkaline earth metal. In one embodiment the metal is calcium.

[0048] When A is cyclic hydrocarbon group, suitable groups include phenylene or fused bicyclic groups such as naphthylene, indenylene, indanylene, bicyclopentadienylene or mixtures thereof. In one embodiment A comprises a benzene ring.

[0049] When A is an acyclic divalent hydrocarbon group, the carbon chain may be linear or branched. In one embodiment A is an acyclic linear hydrocarbon group.

[0050] The overbased sulphonate detergent in one embodiment has a TBN (total base number) of at least 350, in another embodiment at least 400, in another embodiment at least 425, in another embodiment at least 450 and in another embodiment at least 475. In one embodiment the overbased sulphonate detergent has a TBN of 400 or 500.

[0051] The sulphonate detergent is present in one embodiment at 0.1 to 35, in another embodiment 2 to 30, in another embodiment 5 to 25 and in another embodiment 10 to 25 weight percent of the lubricating composition.

45 Overbasing the Sulphonate Detergent

[0052] In one embodiment the sulphonate detergent is overbased. Overbased materials, otherwise referred to as overbased or superbased salts, are generally single phase, homogeneous Newtonian systems characterised by a metal content in excess of that which would be present for neutralisation according to the stoichiometry of the metal and the particular acidic organic compound reacted with the metal. The overbased materials are prepared by reacting an acidic material (typically an inorganic acid or lower carboxylic acid, often carbon dioxide) with a mixture comprising an acidic organic compound, a reaction medium comprising at least one organic solvent and promoter such as phenol or a mixture of alcohols. A mixture of alcohols typically contains methanol and at least one alcohol with 2 to 7 carbon atoms, and may contain 50-60 mole percent methanol. The acidic organic material (substrate) will normally have a sufficient number of carbon atoms to provide a degree of solubility in oil. The amount of excess metal is commonly expressed in terms of metal to substrate ratio. The term "metal to substrate ratio" or "metal ratio" is the ratio of the total equivalents of the metal to the equivalents of the substrate. An overbased sulphonate detergent in one embodiment has a metal ratio of 12.5:1

to 40:1, in another embodiment 13.5:1 to 40:1, in another embodiment 14.5:1 to 40:1, in another embodiment 15.5:1 to 40:1 and in another embodiment 16.5:1 to 40:1. Furthermore the overbased detergent often has a low in-process viscosity and a low final viscosity.

[0053] A sulphonate detergent with 500 TBN and its preparation are disclosed in U.S. Patent 5,792,732. In Example 2 thereof, a 500 TBN all-linear alkylbenzene sulphonate is prepared by reacting an alkyl benzene sulphonate from Witco Corp. (now known as Crompton) with $\text{Ca}(\text{OH})_2$ and CaO in n-heptane and methanol and bubbling with CO_2 . It is also reported in the aforementioned patent (col. 5) that a 500 TBN overbased sulphonate containing highly branched alkylbenzene sulphonate is available from Witco Corp. (now known as Crompton) as Petronate® C-500. Another method for preparing an overbased sulphonate detergent of high metal ratio is disclosed in U.S. Patent 6,444,625 (see, for instance, column 3, bottom). The latter process includes providing a sulphonic acid to a reactor, adding a lime reactant for neutralization and overbasing, adding a lower aliphatic C_1 to C_4 alcohol and a hydrocarbon solvent, and carbonating the process mixture with carbon dioxide during which process the exotherm of the reaction is maintained between 27°C and 57°C . Alternatively, a high metal-ratio detergent may be prepared by using a mixture of short chain alcohols, with or without a hydrocarbon solvent, conducting the addition of lime reactants and carbon dioxide in multiple iterations, and, if desired the process of adding lime and carbon dioxide and of removal of volatile materials may be repeated. The overbased sulphonate detergent in the present invention may be used alone or with other overbased sulphonates. In one embodiment the sulphonate detergent is in a mixture with other sulphonate detergents. Alternatively a 500 TBN sulphonate detergent may be prepared by the process shown in Preparative Example S-1.

Preparative Example S-1

[0054] A sample of a 500 TBN sulphonate detergent is prepared using a vessel with flange and clip, overhead stirrer with paddle and polytetrafluoroethylene (PTFE) stirrer gland, Dean Stark trap and double surface condenser, a mantle/thermocouple temperature controller system, the equipment from just above the mantle to just below the condenser being covered with glass wool. The vessel is charged with 35.1 parts by weight of $\text{C}_{16}\text{-C}_{24}$ alkylbenzene sulphonic acid and 31.8 parts by weight of mineral oil (SN 150) and heated to 30°C . The reactor is charged through a port with 11.6 parts by weight of alcohols containing methanol and a mixture of iso-butanol/amyl alcohol. The weight ratio of methanol to the mixture of iso-butanol/amyl alcohol is 1.31. The reactor is charged with 14.9 parts by weight of calcium hydroxide and the mixture is heated to 54°C at which temperature carbon dioxide is added to form a carbonated product. The carbonated product is further treated three more times with similar (or equal) portions of calcium hydroxide and carbon dioxide. Water is removed by stripping before repeating the addition of alcohol, calcium hydroxide and carbon dioxide two times. The product is stripped and filtered.

[0055] Optionally the lubricating composition further includes a phenate detergent. The phenate detergent is known and includes neutral and overbased metal salts of a sulphur-containing phenate, a non-sulphurised phenate or mixtures thereof. Suitable metal salts are the same as those described for the sulphonate detergent.

[0056] The phenate detergent in one embodiment has a TBN from 30 to 450, in another embodiment 30 to 350 or 290, in another embodiment 40 to 265, in another embodiment 50 to 190 and in another embodiment 70 to 175. In one embodiment the sulphur containing phenate detergent has a TBN of 150, in another embodiment a TBN of 225 and in another embodiment 250.

[0057] Other suitable detergent compounds include a salicylate, carboxylate, phosphate, salixarate or mixtures thereof.

Dispersant

[0058] The invention optionally further includes a dispersant. The dispersant is known and includes an ash-containing dispersant or an ashless-type dispersant, "ashless" dispersant being so named because, prior to mixing with other components of the lubricant, they do not contain metals which form sulfated ash. After admixture, of course, they may acquire metal ions from other components; but they are still commonly referred to as "ashless dispersants." The dispersant may be used alone or in combination with other dispersants. In one embodiment the ashless dispersant does not contain ashforming metals. Ashless type dispersants are characterised by a polar group attached to a relatively high molecular weight hydrocarbon chain. Typical ashless dispersants include a N-substituted long chain hydrocarbon succinimide such as alkenyl succinimide. Examples of N-substituted long chain alkenyl succinimides include polyisobutylene succinimide with number average molecular weight of the polyisobutylene substituent in one embodiment from 350 to 5000, and in another embodiment 500 to 3000. Succinimide dispersants and their preparation are disclosed, for instance in US Patent 4,234,435. Succinimide dispersants are typically the imide formed from a polyamine, typically a poly(ethyleneamine).

[0059] In one embodiment the invention the dispersant is derived from polyisobutylene, an amine and zinc oxide to form a polyisobutylene succinimide complex with zinc.

[0060] In one embodiment the dispersant is derived from half ester, ester or salts of a long chain hydrocarbon acylating agent such as long chain alkenyl succinic acid/anhydride.

[0061] Another class of ashless dispersant is Mannich bases. Mannich dispersants are the reaction products of alkyl phenols with aldehydes (especially formaldehyde) and amines (especially polyalkylene polyamines). The alkyl group typically contains at least 30 carbon atoms.

[0062] The dispersant may also be post-treated by conventional methods by a reaction with any of a variety of agents. Among these are urea, thiourea, dimercaptothiadiazole or derivatives thereof, carbon disulphide, aldehydes, ketones, carboxylic acids, hydrocarbon-substituted succinic anhydrides, maleic anhydride, acrylonitrile, epoxides, boron compounds, and phosphorus compounds.

[0063] In one embodiment of the invention the dispersant is borated using a variety of agents selected from the group consisting of the various forms of boric acid (including metaboric acid, HEO_2 , orthoboric acid, H_3BO_3 , and tetraboric acid, $\text{H}_2\text{B}_4\text{O}_7$), boric oxide, boron trioxide, and alkyl borates.

[0064] The borated dispersant may be prepared by blending the boron compound and the N-substituted long chain alkenyl succinimides and heating them at a suitable temperature in one embodiment from 80°C to 250°C, in another embodiment 90°C to 230°C and in another embodiment 100°C to 210°C, until the desired reaction has occurred. The molar ratio of the boron compounds to the N-substituted long chain alkenyl succinimides is typically 10:1 to 1:4, in another embodiment 4:1 to 1:3, and in another embodiment about 1:2. An inert liquid may be used in performing the reaction. The liquid may include toluene, xylene, chlorobenzene, dimethylformamide and mixtures thereof.

[0065] Other performance additives may be used, such as an antioxidant including a diphenylamine, a hindered phenol, a molybdenum dithiocarbamate, a sulphurised olefin and mixtures thereof (in one embodiment the lubricating composition is free of an antioxidant); corrosion inhibitors including octylamine octanoate; condensation products of dodecyl succinic acid or anhydride and a fatty acid such as oleic acid with a polyamine; metal deactivators including derivatives of benzotriazoles, 1,2,4-triazoles, benzimidazoles, 2-alkyldithiobenzimidazoles or 2-alkyldithiobenzothiazoles; foam inhibitors including copolymers of ethyl acrylate and 2-ethylhexylacrylate and optionally vinyl acetate; demulsifiers including trialkyl phosphates, polyethylene glycols, polyethylene oxides, polypropylene oxides and (ethylene oxide-propylene oxide) polymers; pour point depressants including esters of maleic anhydride-styrene, polymethacrylates, polyacrylates or polyacrylamides; and friction modifiers including fatty acid derivatives such as amines, esters, epoxides, fatty imidazolines, condensation products of carboxylic acids and polyalkylenepolyamines and amine salts of alkylphosphoric acids.

Process

[0066] The invention also includes a process to prepare a lubricating composition, comprising mixing:

- (a) a 2 mm²/s to 12 mm²/s light neutral base oil;
- (b) a heavy neutral base oil or a brightstock with a viscosity above 12 mm²/s to 40 mm²/s; and
- (c) at least one performance additive comprising: a polymeric thickener, a dispersant, an antiwear agent, a detergent or mixtures thereof;

wherein the polymeric thickener has a weight average molecular weight (Mw) of more than 8000 and is present in an amount to provide a total viscosity of the composition of 12 mm²/s or 15 mm²/s to 29 mm²/s; the dispersant or detergent when present imparts basicity to said lubricating composition; and the composition has a total base number of at least 10 mg KOH/g.

[0067] The mixing conditions include a temperature in one embodiment from 15°C to 130°C, in another embodiment 20°C to 120°C and in another embodiment 25°C to 110°C; and for a period of time in one embodiment from 30 seconds to 48 hours, in another embodiment 2 minutes to 24 hours, and in other embodiments 5 minutes to 16 hours or 20 minutes to 4 hours; and at pressures in one embodiment from 86 kPa to 270 kPa (650 mm Hg to 2000 mm Hg), in another embodiment 92 kPa to 200 kPa (690 mm Hg to 1500 mm Hg), and in another embodiment 95 kPa to 130 kPa (715 mm Hg to 1000 mm Hg).

[0068] The process optionally includes mixing other performance additives as described above. The optional performance additives may be added sequentially, separately or as a concentrate. In one embodiment the composition is a concentrate.

Industrial Application

[0069] The lubricating composition of the present invention is useful for an internal combustion engine, for example stationary combustion engine, such as a power station combustion engine; a diesel fuelled engine, a gasoline fuelled engine, a natural gas fuelled engine or a mixed gasoline/alcohol fuelled engine. In one embodiment the internal combustion engine is a 4-stroke and in another embodiment a 2-stroke engine. In one embodiment the diesel fuelled engine is a marine diesel engine.

[0070] In one embodiment of the invention provides a method for lubricating an internal combustion engine, comprising

supplying thereto a lubricant comprising the composition as described herein. The use of the composition may impart one or more of TBN control, cleanliness properties, antiwear performance and deposit control.

[0071] In one embodiment the invention provides a method for lubricating a 2-stroke marine diesel cylinder comprising: lubricating said 2-stroke marine diesel cylinder with a lubricating composition comprising:

- (a) a 2 mm²/s to 12 mm²/s light neutral base oil;
- (b) 0 to 20 weight percent of a heavy neutral base oil or a brightstock with a viscosity above 12 mm²/s to 40 mm²/s;
- (c) a polymeric thickener; and
- (d) at least one performance additive comprising: a dispersant, an antiwear agent, a detergent or mixtures thereof;

wherein the polymeric thickener has a weight average molecular weight (Mw) of more than 8000 and is present in an amount to provide a total viscosity of the lubricating composition of 12 mm²/s or 15 mm²/s to 29 mm²/s (or viscosity grade ranging from SAE 40 to SAE 60); the dispersant or detergent when present imparts basicity to said lubricating composition; and the composition has a total base number of at least 10 mg KOH/g.

[0072] The following examples provide an illustration of the invention. These examples are non exhaustive and are not intended to limit the scope of the invention.

EXAMPLES

[0073] All additive components used in Preparative Examples 1-7 are commercially available from The Lubrizol Corporation. All performance additives used in the examples quote the weight percent as supplied, including the conventional amount of diluent oil.

Preparative Example 1: System Oil Composition (P1)

[0074] A system oil is prepared having a TBN of 1-15, containing 1 wt % dispersant, 0.5 wt % ZDDP, 6 wt % of 250 TBN phenate detergent, 0.5 wt % of neutral sulphonate detergent and base oil containing light solvent neutral oil and brightstock. The system oil composition has a viscosity of 11 mm²/s.

Preparative Example 2: System Oil Composition (P2)

[0075] A System Oil is prepared having a TBN of 2-7, containing 0.5 wt % dispersant, 0.3 wt % ZDDP, 1.0 wt % of 250 TBN phenate detergent, 1.2 wt % of neutral sulphonate detergent, 0.2 wt % of an anti-oxidant and base oil containing 4.8 wt % of Exxon 150 light neutral base oil and 92.2 wt % of Exxon 600 light neutral base stock. The system oil composition has a viscosity of 11 mm²/s.

Preparative Example 3: SAE 50 Lubricating Oil Composition (P3)

[0076] A core lubricating oil composition is prepared having a TBN of 3-30, containing 1.5 wt % dispersant, 0.5 wt % of ZDDP, 12 wt % of 250 TBN phenate, and base oil containing solvent neutral oil and brightstock. The core lubricating oil composition has a viscosity of 19.5 mm²/s.

Preparative Example 4: SAE 50 Lubricating Oil Composition (P4)

[0077] A core lubricating oil composition is prepared having a TBN of 4-10, containing 1 wt % of borated dispersant, 6.5 wt % of 250 TBN phenate, and base oil containing solvent neutral oil and brightstock. The core lubricating oil composition has a viscosity of 19.5 mm²/s.

Preparative Example 5: High TBN Composition (P5)

[0078] A high TBN composition is prepared from a 200 TBN concentrate of 50 wt % of 400 TBN overbased sulphonate detergent and 50 wt % of 500 SN basestock.

Preparative Example 6: High TBN Composition (P6)

[0079] A high TBN composition is prepared from a 400 TBN concentrate of 80 wt % of 500 TBN overbased sulphonate detergent and 20 wt % of 500 SN basestock.

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Preparative Example 7: Polymeric Thickener Composition (P7)

[0080] Preparative Example 7 is a polymethacrylate viscosity index improver with a weight average molecular weight, Mw of 10^5 to 10^6 .

Examples 1-10

[0081] Examples 1-10 are prepared by blending portions of preparative examples 1-7 as shown:

Weight Percentage of Preparative Examples 1-7

[0082]

	Example	TBN	P1	P2	P3	P4	P5	P6	P7
15	EX1	40			94		6		
	EX2	40			97.3			2.7	
	EX3	40				84	16		
	EX4	40				92		8	
20	EX5	40	90.3					6.7	3
	EX6	70			76.5		23.5		
	EX7	70			89			11	
	EX8	70				68	32		
	EX9	70				84.5		15.5	
25	EX10	70	82.5					14.5	3

Example 11 (EX11)

[0083] A lubricating composition is prepared from 79.4 wt % of fresh system oil from Preparative Example 2, 2.6 wt % of 150 TBN phenate detergent, 15 wt % of 400 TBN sulphonate detergent, 0.8 wt % of borated dispersant and 2.2 wt % of the product of Preparative Example 7. The lubricating composition has TBN of 67.2, and a viscosity of 18.1 mm²/s.

Example 12 (EX12)

[0084] A lubricating composition is prepared with 40.3 wt % of fresh system oil from Preparative Example 2, 40.3 wt % of used system oil containing contaminants from an engine stuffing box (a stuffing box is fitted to a crankcase to separate system oil from used cylinder oil) from Preparative Example 2. The used system oil is originally derived from the system oil of Preparative Example 2. The lubricating composition further contains 2.5 wt % of 150 TBN phenate detergent, 14.2 wt % of 400 TBN sulphonate detergent, 0.7 wt % of borated dispersant and 2 wt % of the product of Preparative Example 7. The lubricating composition has TBN of 67.2, and a viscosity of 18.1 mm²/s.

Example 13 (EX13)

[0085] A lubricating composition is prepared with 81.7 wt % of used system oil containing contaminants from an engine stuffing box. The used system oil is originally derived from the system oil of Preparative Example 2. The lubricating composition further contains 2.3 wt % of 150 TBN phenate detergent, 13.4 wt % of 400 TBN sulphonate detergent, 0.7 wt % of borated dispersant and 1.9 wt % of the product of Preparative Example 7. The lubricating composition has TBN of 66.2, and a viscosity of 17.8 mm²/s.

Reference Example 1 (RF1)

[0086] Reference Example is the same as Example 11, except the compound of preparative Example 7 is absent. Furthermore the system oil is replaced with 55 wt % of Exxon 600N base oil and 23.7 wt % of Exxon 150 brightstock base oil. The Reference Examples has TBN of 68.9, and a viscosity of 18.7 mm²/s.

Reference Example 2 (RF2)

[0087] Reference Example is the same as Example 11, except the compound of preparative Example 7 is absent and ZDDP is present at 0.5 wt %. Furthermore the system oil is replaced with 55.9 wt % of Exxon 600N base oil and 23.8 wt % of Exxon 150 brightstock base oil. The Reference Examples has TBN of 68.9, and a viscosity of 18.7 mm²/s.

Test 1: Cameron Plint Wear Test

[0088] The Cameron Plint TE-77™ is a reciprocating wear tester. In this test a steel ball upper specimen is reciprocated against a steel flat lower specimen. The lubricant sample is initially treated with 3.5 wt % sulphuric acid. The Cameron Plint is then charged with 10 ml of the sample and heated to 50°C and held for 20 minutes. The sample is then subject to a load of 240 N over two minutes while at the same time reciprocation is started at 10 Hz over 15 mm stroke length. The sample is then heated to 350°C at 2°C per minute and then held at temperature for 3 hours. At the end of the test the onset of film failure is measured. The onset of film failure is determined by the temperature at which the oil film as measured by the contact potential first falls to 80 % of its starting value. The results obtained by testing the lubricants of the indicated Examples are as shown:

Example	Temperature of Onset of Film Failure (°C)
EX11	320
EX12	300
EX13	290
RF1	250
RF2	240

[0089] The analysis of the experimental data obtained indicates that presence of the additive package of the invention provides one or more of TBN control, cleanliness properties, antiwear performance and deposit control to a system oil.

Examples 14 to 26

[0090] Examples 14 to 26 contain 2 wt % of various polymethacrylate polymeric thickeners, 1.5 wt % of a succinimide dispersant, 7 wt % of a brightstock base oil, between 67.4 and 72.3 wt % of a light neutral base oil and a detergent package. The detergent package in Examples 14 to 22 contains a mixture of sulphonate detergents with a TBN ranging from 350 to 500 TBN. Examples 23 to 26 contain a mixture of sulphonate and phenate detergents.

[0091] Reference Example 3 (RF3) is a commercially available marine diesel cylinder lubricant.

Test 2: Panel Coker

[0092] Approximately 233g of sample is placed in a 250 ml Panel Coker apparatus and heated to 325°C. The sample is splashed against a metal plate for 15 seconds and then baked for 45 seconds. The splashing and baking cycle is continued for approximately 3 hours. The sample is cooled to room temperature and the amount of deposits left on the metal plate is weighed. The results obtained by testing the lubricants of the indicated Examples are as shown: .

Example	Deposits (mg)	Example	Deposits (mg)
RF3	121.0	EX20	41.1
EX14	43.2	EX21	78.7
EX15	23.7	EX22	101.8
EX16	77.3	EX23	27.4
EX17	57.7	EX24	55.3
EX18	45.7	EX25	22.6
EX19	106.2	EX26	62.8

[0093] In summary the results of the Panel Coker test demonstrate that the composition of the invention has reduced deposit formation than a conventional marine diesel cylinder lubricant.

[0094] While the invention has been explained, it is to be understood that various modifications thereof will become apparent to those skilled in the art upon reading the specification. Therefore, it is to be understood that the invention

disclosed herein is intended to cover such modifications as fall within the scope of the appended claims.

[0095] Each of the documents referred to above is incorporated herein by reference. Except in the Examples, or where otherwise explicitly indicated, all numerical quantities in this description specifying amounts of materials, reaction conditions, molecular weights, number of carbon atoms, and the like, are to be understood as modified by the word "about." Unless otherwise indicated, each chemical or composition referred to herein should be interpreted as being a commercial grade material which may contain the isomers, by-products, derivatives, and other such materials which are normally understood to be present in the commercial grade. However, the amount of each chemical component is presented exclusive of any solvent or diluent oil, which may be customarily present in the commercial material, unless otherwise indicated. It is to be understood that the upper and lower amount, range, and ratio limits set forth herein may be independently combined. Similarly, the ranges and amounts for each element of the invention may be used together with ranges or amounts for any of the other elements. As used herein any member of a genus (or list) may be excluded from the claims.

[0096] As used herein, the term "hydrocarbyl substituent" or "hydrocarbyl group" is used in its ordinary sense, which is well-known to those skilled in the art. Specifically, it refers to a group having a carbon atom directly attached to the remainder of the molecule and having predominantly hydrocarbon character. Examples of hydrocarbyl groups include:

(i) hydrocarbon substituents, that is, aliphatic (e.g., alkyl or alkenyl), alicyclic (e.g., cycloalkyl, cycloalkenyl) substituents, and aromatic-, aliphatic-, and alicyclic-substituted aromatic substituents, as well as cyclic substituents wherein the ring is completed through another portion of the molecule (e.g., two substituents together form a ring);

(ii) substituted hydrocarbon substituents, that is, substituents containing non-hydrocarbon groups which, in the context of this invention, do not alter the predominantly hydrocarbon nature of the substituent (e.g., halo (especially chloro and fluoro), hydroxy, alkoxy, mercapto, alkylmercapto, nitro, nitroso, and sulfoxy);

(iii) hetero substituents, that is, substituents which, while having a predominantly hydrocarbon character, in the context of this invention, contain other than carbon in a ring or chain otherwise composed of carbon atoms. Heteroatoms include sulfur, oxygen, nitrogen, and encompass substituents as pyridyl, furyl, thienyl and imidazolyl. In general, no more than two, or no more than one, non-hydrocarbon substituent will be present for every ten carbon atoms in the hydrocarbyl group; typically, there will be no non-hydrocarbon substituents in the hydrocarbyl group.

[0097] It is known that some of the materials described above may interact in the final formulation, so that the components of the final formulation may be different from those that are initially added. The products formed thereby, including the products formed upon employing the composition of the present invention in its intended use, may not be susceptible of easy description. Nevertheless, all such modifications and reaction products are included within the scope of the present invention; the present invention encompasses the composition prepared by admixing the components described above.

[0098] Various preferred features and embodiments of the present invention will now be described with reference to the following numbered paragraphs (paras)

1. A method of lubricating an internal combustion engine with a power output of at least 1600 kilowatts, with a lubricating composition, the method comprising:

- (1) monitoring one or more performance characteristics of the engine;
- (2) selecting an additive package to provide a desired TBN level to a lubricating composition to modify the performance characteristics of the engine;
- (3) combining the additive package of step (2) with (a) a component having a viscosity of about 2 mm²/s to 12 mm²/s, comprising a light neutral base oil, and optionally (b) a component having a viscosity of above 12 mm²/s to about 40 mm²/s, comprising heavy neutral base oil or a brightstock, to form a lubricating composition; and
- (4) supplying the lubricating composition of step (3) to the engine;

wherein the lubricating composition has a total base number of at least 10 mg KOH/g; and wherein the component comprising the light neutral base oil is supplied from an oil reservoir available to said internal combustion engine.

2. The method of para 1, wherein the total base number of the lubricating composition is about 30 or higher.

3. The method of para 1, wherein the internal combustion engine has a power output of at least about 3000 kilowatts.

4. The method of para 1, wherein the internal combustion engine is a 2-stroke marine diesel engine.

5. The method of para 1, wherein the light neutral base oil is provided from a new or used crankcase system oil.

6. The method of para 1, wherein the total base number of the lubricating composition is 30 mg KOH/g or higher.
7. The method of para 1, wherein the one or more characteristics of the engine comprise wear, engine load, variation in TBN, deposits, or corrosion.
8. The method of para 1, wherein said reservoir is a day tank or cylinder oil service tank or a storage tank.
9. The method of para 1, wherein the additive package of step (2) is combined with a base oil in a blender.
10. The method of para 1 wherein the additive package of step (2) further comprises at least one performance additive comprising a polymeric thickener, a dispersant, an antiwear agent, a detergent or mixtures thereof.
11. The method of para 10, wherein the polymeric thickener comprises polyisobutylene with a weight average molecular weight Mw of more than about 8000 to about 25,000 or a poly(meth)acrylate with a weight average molecular weight Mw of about 20,000 to about 300,000.
12. The method of para 10, wherein the detergent comprises a sulphonate, a phenate or mixtures thereof.
13. The method of para 10, wherein the dispersant comprises a polyisobutylene succinimide.
14. The method of para 10, wherein the antiwear agent comprises a metal hydrocarbyl dithiophosphate.
15. The method of para 1, wherein the light neutral base oil is present at about 50 to about 99.9 weight percent of the lubricating composition; the heavy neutral base oil or brightstock is present at about 0.01 to about 25 weight percent of the lubricating composition; and at least one performance additive is present at about 0.01 to about 25 weight percent of the lubricating composition.
16. The use of a system oil additised with an additive package to make a cylinder lubricant.
17. The use of the system oil of para 16, as a cylinder lubricant in a 2-stroke marine diesel engine.
18. The use of the system oil of para 16, wherein the system oil is from used crankcase system oil.

Claims

1. A method of lubricating an internal combustion engine with a power output of at least 1600 kilowatts, with a lubricating composition, the method comprising:
 - (1) monitoring one or more performance characteristics of the engine;
 - (2) selecting an additive package to provide a desired TBN level to a lubricating composition to modify the performance characteristics of the engine, wherein the additive package comprises a phenate detergent with a TBN of 30 to 450;
 - (3) combining the additive package of step (2) with (a) a component having a viscosity of about 2 mm²/s to 12 mm²/s, comprising a light neutral base oil, wherein the light neutral base oil is provided from a new or used crankcase system oil, and optionally (b) a component having a viscosity of above 12 mm²/s to about 40 mm²/s, comprising heavy neutral base oil or a brightstock, to form a lubricating composition; and
 - (4) supplying the lubricating composition of step (3) to the engine;wherein the lubricating composition has a total base number of at least 10 mg KOH/g; and wherein the component comprising the light neutral base oil is supplied from an oil reservoir available to said internal combustion engine.
2. The method of claim 1, wherein the internal combustion engine is a 2-stroke marine diesel engine.
3. The method of claim 1, wherein the total base number of the lubricating composition is about 30 or higher.
4. The method of claim 1, wherein the one or more characteristics of the engine comprise wear, engine load, variation in TBN, deposits, or corrosion.

5. The method of claim 1, wherein the additive package of step (2) is combined with a base oil in a blender.
6. The method of claim 1 wherein the additive package of step (2) further comprises at least one performance additive comprising a polymeric thickener, a dispersant, an antiwear agent, a detergent or mixtures thereof.
7. The method of claim 6, wherein the polymeric thickener comprises polyisobutylene with a weight average molecular weight Mw of more than about 8000 to about 25,000 or a poly(meth)acrylate with a weight average molecular weight Mw of about 20,000 to about 300,000.
8. The method of claim 1, wherein the light neutral base oil is present at 50 to 99.9 weight percent of the lubricating composition; the heavy neutral base oil or brightstock is present at 0.01 to 25 weight percent of the lubricating composition; and at least one performance additive is present at 0.01 to 25 weight percent of the lubricating composition.
9. The method of claim 1, wherein the phenate includes neutral and overbased metal salts of a sulphur-containing phenate, a non-sulphurised phenate or mixtures thereof.
10. The method of claim 1, wherein the phenate has a TBN of 70 to 175 mg KOH/g.



EUROPEAN SEARCH REPORT

Application Number
EP 10 17 9036

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**ANNEX TO THE EUROPEAN SEARCH REPORT
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