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Lubricating oil additive and lubricating oil composition containing the same.

㉚ Fuel consumption in an internal combustion engine is reduced by contacting the moving surfaces thereof with a lubricating oil having present therein a complex obtained by reacting a borated alkyl catechol with an oil-soluble alkyl or alkenyl mono or bis succinimide.

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1 LUBRICATING OIL ADDITIVE AND
 LUBRICATING OIL COMPOSITION CONTAINING THE SAME

 This invention relates to lubricating oil additives
which serve to reduce friction in internal combustion
5 engines and to the use of such additives in lubricant
compositions.

 With the crisis associated with diminishing
amounts of fossil fuel and the rapidly increasing prices
for this fuel, there has been a great deal of interest in
10 reducing the amount of fuel consumed by automobile
engines, and the like.

 Thus, there is a great need to find lubricants
that reduce the overall friction in the engine, thus
15 reducing the energy requirements thereto.

 U.S. Patent No. 2,795,548 discloses the use of
lubricating oil compositions containing borated alkyl
catechols. The oil compositions are used in the crankcase
of an internal combustion engine in order to reduce oxi-
20 dation of the oil and corrosion of the metal parts of the
engine.

 There is a problem with the use of borated alkyl
catechols in lubricating oils since they are sensitive to
moisture and hydrolyze readily. The hydrolysis leads to
haze and/or precipitate formation which must be filtered
25 out prior to use.

 It has now been found that the borated alkyl
catechols may be stabilized against hydrolysis by reacting
the borated alkyl catechol with an alkenyl or alkyl
30 mono or bis succinimide, and that lubricating the crankcase
of an internal combustion engine with a lubricating oil
containing such a reaction product reduces the fuel
consumption of the engine.

 Thus in accordance with one aspect of the present
invention, there is provided a lubricating oil additive
35 comprising a complex obtainable by reacting a borated

1 alkyl catechol with an oil soluble alkyl or alkenyl
mono or bis succinimide.

In accordance with another aspect of the invention,
there is provided a lubricating oil composition
5 comprising an oil of lubricating viscosity and, in an
amount effective to reduce friction, an additive prepared
by reacting a borated alkyl catechol with an oil soluble
alkyl or alkenyl mono or bis succinimide.

Other additives may also be present in the
10 lubricating oil in order to obtain a proper balance of
properties such as dispersancy, corrosion, wear and oxi-
dation inhibition which are critical for the proper opera-
tion of an internal combustion engine.

In accordance with a further aspect of the invention, there
15 is provided a method of reducing the fuel consumption of an
internal combustion engine comprising treating the moving surface
thereof with the lubricating oil composition as described
above. Specifically, improvements in fuel mileage of from
1% to 2% may be obtained by employing the composition of
20 this invention. This fuel economy improvement can be
obtained in both compression-ignition engines, that is,
diesel engines, and spark-ignition engines, that is,
gasoline engines.

Moreover, lubricating oil compositions contain-
25 ing the borated alkyl catechol-succinimide complex of this
invention have been found additionally to possess antioxi-
dant properties and when employed in diesel engines they
possess diesel deposit inhibition.

The complex between the borated alkyl catechol
30 and the alkenyl or alkyl succinimide may be prepared in
situ. That is when borated alkyl catechol and a

1 sufficient amount of an alkenyl or alkyl mono or bis succinimide to
stabilize the borated alkyl catechol against hydrolysis
are added to the lubricating oil, the complex is formed in
situ.

5 In this aspect, the present invention relates to
a lubricating oil composition comprising an oil of lubri-
cating viscosity and an effective amount to reduce fric-
tion of a borated alkyl catechol and an effective amount
of an alkenyl mono or bis succinimide to stabilize the borated alkyl
10 catechol against hydrolysis.

When employed in this manner, other additives
may also be present in the lubricating oil in order to
obtain a proper balance of properties such as dispersion,
corrosion, wear and oxidation which are critical for the
15 proper operation of an internal combustion engine.

Thus, another aspect of the present invention is
a lubricating oil composition especially useful in the
crankcase of an internal combustion engine for the purpose
of improving the fuel consumption of said engine
20 comprising

(a) an oil of lubricating viscosity;
and

- (b) an effective amount of each of the following:
1. an alkenyl succinimide,
 - 25 2. a Group II metal salt of a dihydrocarbyl
dithiophosphoric acid,
 3. a neutral or overbased alkali or alkaline
earth metal hydrocarbyl sulfonate or mixture there-
of,
 - 30 4. a neutral or overbased alkali or alkaline
earth metal alkylated phenate, or mixture thereof,
and
 5. a borated alkyl catechol friction modifier.

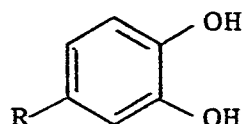
In still another aspect of this invention, there is
35 provided a method of reducing fuel consumption of an
internal combustion engine by treating the moving surfaces
thereof with the lubricating oil composition described
above.

1 The borated alkyl catechols may be prepared by
borating a alkyl catechol with boric acid with removal of
the water of reaction. Preferably, there is sufficient
boron present such that each boron will react with from
5 1.5 to 2.5 hydroxyl groups present in the reaction mix-
ture.

The reaction may be carried out at a temperature
in the range of 60°C to 135°C, in the absence or presence
of any suitable organic solvent, such as methanol, benzene,
10 xylenes, toluene, or neutral oil.

The alkyl catechols or mixtures thereof which
may be used to prepare the borated alkyl catechols used in
this invention are preferably monoalkyl catechols of
Formula I

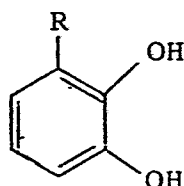
15



I

wherein R is alkyl containing 10 to 30 carbon atoms
20 and preferably from 16 to 26 carbon atoms. Also, up to
25% by weight but preferably less than 10% by weight of
the monoalkyl catechols may have the R group in a position
adjacent or ortho to one of the hydroxy groups and has the
Formula II

25



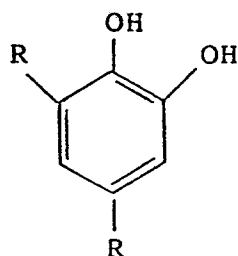
II

30 wherein R is defined above.

Also included among alkyl catechols which may be
used to prepare the borated alkyl catechols of this inven-
tion are dialkyl catechols which are generally of
Formula III

35

1



III

5

wherein R is defined above. Trialkyl catechols may also be employed although they are not preferred.

Among the alkyl catechols which may be employed are decyl catechol, undecyl catechol, dodecyl catechol, tetradecyl catechol, pentadecyl catechol, hexadecyl catechol, octadecyl catechol, eicosyl catechol, hexacosyl catechol, triacontyl catechol, and the like. Also, a mixture of alkyl catechols may be employed such as a mixture of C_{14} to C_{26} alkyl catechols, for example C_{14} - C_{18} alkyl catechols or C_{16} - C_{26} alkyl catechols, may be used.

The alkyl catechols of the Formula III may be prepared by reacting a C_{10} to C_{30} olefin such as a branched olefin or straight-chained alpha-olefin containing 10 to 30 carbon atoms with pyrocatechol in the presence of a sulfonic acid catalyst at a temperature of from about 60°C to 200°C , and preferably 125°C to 180°C in an essentially inert solvent at atmospheric pressure. Examples of the inert solvents include benzene, toluene, chlorobenzene and 250 Thinner which is a mixture of aromatics, paraffins and naphthenes.

The term "branched olefin" means that branching occurs at the double bond. The term "straight chain alpha olefin" means that the alpha olefin contains little (less than 10%) or no branching at the double bond or elsewhere.

A product which is predominantly monoalkyl catechol may be prepared by using molar ratios of reactants and preferably a 10% by weight molar excess of branched olefin or alpha-olefin over catechol is used. When used at molar ratios, the resulting products are generally monoalkyl catechols but contain some amounts of dialkyl catechol. In any event, molar excess of pyrocatechol (i.e., 2 equivalents of pyrocatechol for each equivalent

1 of olefin) can be used in order to enhance monoalkylation
if predominantly monoalkyl catechol is desired. Predomi-
nantly dialkyl catechols may be prepared by employing two
equivalents to pyrocatechol of the same or different
5 olefin.

Use of a branched olefin results in a greater
proportion of alkyl catechols of Formula I than use of
straight-chain alpha-olefins. Use of such branched ole-
fins generally results in greater than 90% alkyl catechol
10 of Formula I and less than 10% alkyl catechol of
Formula II.

The borated alkyl catechols are stabilized
against hydrolysis by reacting the catechols with an alkyl
or alkenyl mono or bis succinimide. In the preferred
15 embodiment, an alkyl or alkenyl mono-succinimide is
employed.

The oil soluble alkenyl or alkyl mono- or
bis-succinimides which are employed in this invention are
generally known as lubricating oil detergents and are
20 described in for example U.S. Patents Nos. 2,992,708 3,018,291,
3,024,237, 3,100,673, 3,219,666, 3,172,892 and 3,272,746.
The alkenyl succinimides are the reaction product of a
polyolefin polymer-substituted succinic anhydride with an
amine, preferably a polyalkylene polyamine. The poly-
25 olefin polymer-substituted succinic anhydrides are
obtained by reaction of a polyolefin polymer or a deriva-
tive thereof with maleic anhydride. The succinic anhy-
dride thus obtained is reacted with the amine compound.
The preparation of the alkenyl succinimides has been
30 described many times in the art. See, for example, U.S.
Patent Nos. 3,390,082, 3,219,666 and 3,172,892.
Reduction of the alkenyl substituted succinic anhydride
yields the corresponding alkyl derivative. A product
comprising predominantly mono- or bis-succinimide can be
35 prepared by controlling the molar ratios of the reactants.
Thus, for example, if one mole of amine is reacted with

1 one mole of the alkenyl or alkyl substituted succinic
anhydride, a predominantly mono-succinimide product will
be prepared. If two moles of the succinic anhydride are
reacted per mole of polyamine, a bis-succinimide will be
5 prepared.

Particularly good results are obtained with the
lubricating oil compositions of this invention when the
alkenyl succinimide is a mono-succinimide prepared from a
polyisobutene-substituted succinic anhydride of a poly-
10 alkylene polyamine.

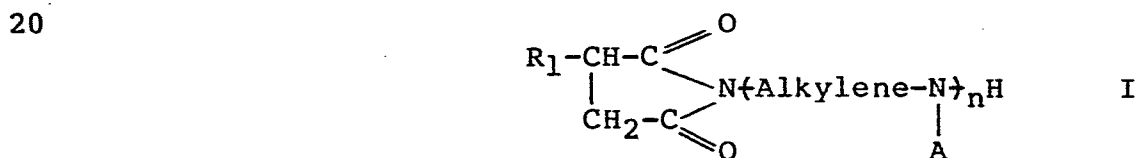
The polyisobutene from which the polyisobutene-
substituted succinic anhydride is obtained by polymerizing
isobutene and can vary widely in its compositions. The
average number of carbon atoms can range from 30 or less
15 to 250 or more, with a resulting number average molecular
weight of about 400 or less to 3,000 or more. Preferably,
the average number of carbon atoms per polyisobutene mole-
cule will range from about 50 to about 100 with the poly-
isobutenes having a number average molecular weight of
20 about 600 to about 1,500. More preferably, the average
number of carbon atoms per polyisobutene molecule ranges
from about 60 to about 90, and the number average
molecular weight ranges from about 800 to 1,300. The
polyisobutene is reacted with maleic anhydride according
25 to well-known procedures to yield the polyisobutene-sub-
stituted succinic anhydride. See, for example, U.S.
Patent Nos. 4,388,471 and 4,450,281.

In preparing the alkenyl succinimide, the sub-
stituted succinic anhydride is reacted with a polyalkylene
30 polyamine to yield the corresponding succinimide. Each
alkylene radical of the polyalkylene polyamine usually has
up to about 8 carbon atoms. The number of alkylene radi-
cals can range up to about 8. The alkylene radical is
exemplified by ethylene, propylene, butylene, trimethyl-
35 ene, tetramethylene, pentamethylene, hexamethylene, octa-
methylene, etc. The number of amino groups generally, but
not necessarily, is one greater than the number of
alkylene radicals present in the amine, i.e., if a

1 polyalkylene polyamine contains 3 alkylene radicals, it
will usually contain 4 amino radicals. The number of
amino radicals can range up to about 9. Preferably, the
alkylene radical contains from about 2 to about 4 carbon
5 atoms and all amine groups are primary or secondary. In
this case, the number of amine groups exceeds the number
of alkylene groups by 1. Preferably the polyalkylene
polyamine contains from 3 to 5 amine groups. Specific
examples of the polyalkylene polyamines include ethylene-
10 diamine, diethylenetriamine, triethylenetetramine, propyl-
enediamine, tripropylenetetramine, tetraethylenepentamine,
trimethylenediamine, pentaethylenhexamine, di-(trimethyl-
ene)triamine, tri(hexamethylene)tetramine, etc.

Other amines suitable for preparing the alkenyl
15 succinimide useful in this invention include the cyclic
amines such as piperazine, morpholine and dipiperazines.

Preferably the alkenyl succinimides used in the
compositions of this invention have the following formula:



25 wherein:

a. R_1 represents an alkenyl group, preferably a sub-
stantially saturated hydrocarbon prepared by polymerizing
aliphatic monoolefins. Preferably R_1 is prepared from
isobutene and has an average number of carbon atoms and a
30 number average molecular weight as described above;

b. the "Alkylene" radical represents a substantially
hydrocarbyl group containing up to about 8 carbon atoms
and preferably containing from about 2-4 carbon atoms as
described hereinabove;

35 c. A represents a hydrocarbyl group, an amine-sub-
stituted hydrocarbyl group, or hydrogen. The hydrocarbyl
group and the amine-substituted hydrocarbyl groups are
generally the alkyl and amino-substituted alkyl analogs of

1 the alkylene radicals described above. Preferably A
represents hydrogen;

d. n represents an integer of from about 1 to 10,
and preferably from about 3-5.

5 The alkenyl succinimide is present in the lubri-
cating oil compositions of the invention in an amount
effective to stabilize the borated alkyl catechols against
hydrolysis and to act as a dispersant and prevent the
deposit of contaminants formed in the oil during operation
10 of the engine.

The exact structure of the complex of this
invention is not known. For certain, however, while not
limiting this invention to any theory, it is believed to
be compounds in which boron is either complexed, by or the
15 salt of, one or more nitrogen atoms of the basic nitrogen
contained in the succinimide. Therefore, it is preferred
that the alkenyl succinimide contain at least 2 and pre-
ferably 3-5 basic nitrogens per atoms.

The complex may be formed by reacting the
20 borated alkyl catechol and the succinimide together neat
at a temperature above the melting point of the mixture of
reactants and below the decomposition temperature, or in a
diluent in which both reactants are soluble. For example,
the reactants may be combined in the proper ratio in the
25 absence of a solvent to form a homogeneous product which
may be added to the oil or the reactants may be combined
in the proper ratio in a solvent such as toluene or
chloroform, the solvent stripped off, and the complex thus
formed may be added to the oil. Alternatively, the com-
30 plex may be prepared in a lubricating oil as a concentrate
containing from about 20 to 90% by weight of the complex,
which concentrate may be added in appropriate amounts to
the lubricating oil in which it is to be used or the com-
plex may be prepared directly in the lubricating oil in
35 which it is to be used.

The diluent is preferably inert to the reactants
and products formed and is used in an amount sufficient to

1 insure solubility of the reactants and to enable the mixture to be efficiently stirred.

Temperatures for preparing the complex may be in the range of from 25°C to 200°C and preferably 25°C to 5 100°C depending on whether the complex is prepared neat or in a diluent, i.e., lower temperatures may be used when a solvent is used.

An effective amount of succinimide is added in order to stabilize the borated alkyl catechols against 10 hydrolysis. In general, weight percent ratios of succinimide to borated alkyl catechol used to form the complex are in the range of 3:1 to 16:1 and preferably from 3:1 to 10:1 and most preferably 3:1 to 6:1. This latter ratio is preferred if the complex is made and/or stored neat or in 15 the absence of solvent or lubricating oil and under atmospheric conditions.

As used herein, the term "stabilized against hydrolysis" means that the borated alkyl catechol-succinimide complex does not form a precipitate due to the hydro- 20 lysis of the borated catechol for a period of at least three months when stored at room temperature (about 15-25°C) and ambient humidity.

The amount of the complex required to be effective for reducing friction in lubricating oil compositions 25 may range from 0.5% to 20% by weight percent. However, in the preferred embodiment, it is desirable to add sufficient complex so that the amount of borated catechol is added at a range from 0.1% to about 4% by weight of the total lubricant composition and preferably is present in 30 the range of from 0.2% to 2% by weight and most preferably 0.5% to 1%. The succinimide is present in the complex of the invention in an amount effective to stabilize the borated alkyl catechol against hydrolysis and which allows the borated alkyl catechol to function as effective friction 35 reducing agents.

Also, the succinimide in the complex acts as a dispersant and prevents the deposition of contaminants formed in the oil during operation of the engine.

1 In general, the complexes of this invention may
also be used in combination with other additive systems in
conventional amounts for their known purpose.

For example, for application in modern crankcase
5 lubricants, the base composition described above will be
formulated with supplementary additives to provide the
necessary stability, detergency, dispersancy, anti-wear
and anti-corrosion properties.

Thus, as another embodiment of this invention,
10 the lubricating oils to which the complexes prepared by
reacting the borated alkyl catechols and succinimides may
contain an alkali or alkaline earth metal hydrocarbyl
sulfonate, an alkali or alkaline earth metal phenate, and
Group II metal salt dihydrocarbyl dithiophosphate.

Also, since the succinimides act as excellent
15 dispersants, additional succinimide may be added to the
lubricating oil compositions, above the amounts added in
the form of the complex with the borated alkyl catechols.
The amount of succinimides can range up to about 20% by
20 weight of the total lubricating oil compositions.

The alkali or alkaline earth metal hydrocarbyl
sulfonates may be either petroleum sulfonate, synthetic-
ally alkylated aromatic sulfonates, or aliphatic sul-
fonates such as those derived from polyisobutylene. One
25 of the more important functions of the sulfonates is to
act as a detergent and dispersant. These sulfonates are
well known in the art. The hydrocarbyl group must have a
sufficient number of carbon atoms to render the sulfonate
molecule oil soluble. Preferably, the hydrocarbyl portion
30 has at least 20 carbon atoms and may be aromatic or ali-
phatic, but is usually alkylaromatic. Most preferred for
use are calcium, magnesium or barium sulfonates which are
aromatic in character.

Certain sulfonates are typically prepared by
35 sulfonating a petroleum fraction having aromatic groups,
usually mono- or dialkylbenzene groups, and then forming
the metal salt of the sulfonic acid material. Other feed-
stocks used for preparing these sulfonates include

1 synthetically alkylated benzenes and aliphatic hydrocar-
bons prepared by polymerizing a mono- or diolefin, for
example, a polyisobutenyl group prepared by polymerizing
isobutene. The metallic salts are formed directly or by
5 metathesis using well-known procedures. The sulfonates
may be neutral or overbased having base numbers up to
about 400 or more. Carbon dioxide and calcium hydroxide or
oxide are the most commonly used material to produce the
basic or overbased sulfonates. Mixtures of neutral and
10 overbased sulfonates may be used. The sulfonates are
ordinarily used so as to provide from 0.3% to 10% by
weight of the total composition. Preferably, the neutral
sulfonates are present from 0.4% to 5% by weight of the
total composition and the overbased sulfonates are present
15 from 0.3% to 3% by weight of the total composition.

The phenates for use in this invention are those
conventional products which are the alkali or alkaline
earth metal salts of alkylated phenols. One of the func-
tions of the phenates is to act as a detergent and disper-
20 sant. Among other things, it prevents the deposit of
contaminants formed during high temperature operation of
the engine. The phenols may be mono- or polyalkylated.

The alkyl portion of the alkyl phenate is
present to lend oil solubility to the phenate. The alkyl
25 portion can be obtained from naturally occurring or syn-
thetic sources. Naturally occurring sources include
petroleum hydrocarbons such as white oil and wax. Being
derived from petroleum, the hydrocarbon moiety is a mix-
ture of different hydrocarbonyl groups, the specific compo-
30 sition of which depends upon the particular oil stock
which was used as a starting material. Suitable synthetic
sources include various commercially available alkenes and
alkane derivatives which, when reacted with the phenol,
yield an alkylphenol. Suitable radicals obtained include
35 butyl, hexyl, octyl, decyl, dodecyl, hexadecyl, eicosyl,
triacontyl, and the like. Other suitable synthetic sour-
ces of the alkyl radical include olefin polymers such as
polypropylene, polybutylene, polyisobutylene and the like.

1 The alkyl group can be straight-chained or
branch-chained, saturated or unsaturated (if unsaturated,
preferably containing not more than 2 and generally not
more than 1 site of olefinic unsaturation). The alkyl
5 radicals will generally contain from 4 to 30 carbon atoms.
Generally when the phenol is monoalkyl-substituted, the
alkyl radical should contain at least 8 carbon atoms. The
phenate may be sulfurized if desired. It may be either
neutral or overbased and if overbased will have a base
10 number of up to 200 to 300 or more. Mixtures of neutral
and overbased phenates may be used.

The phenates are ordinarily present in the oil
to provide from 0.2% to 27% by weight of the total compo-
sition. Preferably, the neutral phenates are present from
15 0.2% to 9% by weight of the total composition and the
overbased phenates are present from 0.2 to 13% by weight
of the total composition. Most preferably, the overbased
phenates are present from 0.2% to 5% by weight of the
total composition. Preferred metals are calcium, magne-
20 sium, strontium or barium.

The sulfurized alkaline earth metal alkyl
phenates are preferred. These salts are obtained by a
variety of processes such as treating the neutralization
product of an alkaline earth metal base and an alkylphenol
25 with sulfur. Conveniently the sulfur, in elemental form,
is added to the neutralization product and reacted at
elevated temperatures to produce the sulfurized alkaline
earth metal alkyl phenate.

If more alkaline earth metal base were added
30 during the neutralization reaction than was necessary to
neutralize the phenol, a basic sulfurized alkaline earth
metal alkyl phenate is obtained. See, for example, the
process of Walker et al, U.S. Patent No. 2,680,096. Addi-
tional basicity can be obtained by adding carbon dioxide
35 to the basic sulfurized alkaline earth metal alkyl
phenate. The excess alkaline earth metal base can be
added subsequent to the sulfurization step but is

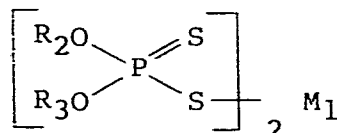
1 conveniently added at the same time as the alkaline earth
metal base is added to neutralize the phenol.

Carbon dioxide and calcium hydroxide or oxide
are the most commonly used material to produce the basic
5 or "overbased" phenates. A process wherein basic sulfur-
ized alkaline earth metal alkylphenates are produced by
adding carbon dioxide is shown in Hanneman, U.S. Patent
No. 3,178,368.

The Group II metal salts of dihydrocarbyl
10 dithiophosphoric acids exhibit wear, antioxidant and ther-
mal stability properties. Group II metal salts of phos-
phorodithioic acids have been described previously. See,
for example, U.S. Patent No. 3,390,080, columns 6 and 7,
wherein these compounds and their preparation are
15 described generally. Suitably, the Group II metal salts
of the dihydrocarbyl dithiophosphoric acids useful in the
lubricating oil composition of this invention contain from
about 4 to about 12 carbon atoms in each of the hydro-
carbyl radicals and may be the same or different and may
20 be aromatic, alkyl or cycloalkyl. Preferred hydrocarbyl
groups are alkyl groups containing from 4 to 8 carbon
atoms and are represented by butyl, isobutyl, sec.-butyl,
hexyl, isohexyl, octyl, 2-ethylhexyl and the like. The
metals suitable for forming these salts include barium,
25 calcium, strontium, zinc and cadmium, of which zinc is
preferred.

Preferably, the Group II metal salt of a
dihydrocarbyl dithiophosphoric acid has the following
formula:

30



35 wherein:

e. R_2 and R_3 each independently represent hydro-
carbyl radicals as described above, and

- 1 f. M_1 represents a Group II metal cation as described above.

 The dithiophosphoric salt is present in the lubricating oil compositions of this invention in an amount effective to inhibit wear and oxidation of the lubricating oil. The amount ranges from about 0.1 to about 4 percent by weight of the total composition, preferably the salt is present in an amount ranging from about 0.2 to about 2.5 percent by weight of the total lubricating oil composition. The final lubricating oil composition will ordinarily contain 0.025 to 0.25% by weight phosphorus and preferably 0.05 to 0.15% by weight.

 The finished lubricating oil may be single or multigrade. Multigrade lubricating oils are prepared by adding viscosity index (VI) improvers. Typical viscosity index improvers are polyalkyl methacrylates, ethylene propylene copolymers, styrene diene copolymers and the like. So-called decorated VI improvers having both viscosity index and dispersant properties are also suitable for use in the formulations of this invention.

 The lubricating oil used in the compositions of this invention may be mineral oil or in synthetic oils of lubricating viscosity and preferably suitable for use in the crankcase of an internal combustion engine. Crankcase lubricating oils ordinarily have a viscosity of about 1300 cst 0°F to 22.7 cst at 210°F (99°C). The lubricating oils may be derived from synthetic or natural sources. Mineral oil for use as the base oil in this invention includes paraffinic, naphthenic and other oils that are ordinarily used in lubricating oil compositions. Synthetic oils include both hydrocarbon synthetic oils and synthetic esters. Useful synthetic hydrocarbon oils include liquid polymers of alpha olefins having the proper viscosity. Especially useful are the hydrogenated liquid oligomers of C₆-12 alpha olefins such as 1-decene trimer. Likewise, alkyl benzenes of proper viscosity such as didodecyl benzene, can be used. Useful synthetic esters include the esters of both monocarboxylic acid and polycarboxylic

1 acids as well as monohydroxy alkanols and polyols.
Typical examples are didodecyl adipate, pentaerythritol
tetracaprate, di-2-ethylhexyl adipate, dilaurylsebacate
and the like. Complex esters prepared from mixtures of
5 mono and dicarboxylic acid and mono and dihydroxy alkanols
can also be used.

Blends of hydrocarbon oils with synthetic oils
are also useful. For example, blends of 10 to 25 weight
percent hydrogenated 1-decene trimer with 75 to 90 weight
10 percent 150 SUS (100°F) mineral oil gives an excellent
lubricating oil base.

Additive concentrates are also included within
the scope of this invention. They usually include from
about 90 to 20 weight percent of an oil of lubricating
15 viscosity and from about 20 to 90 weight percent of the
complex additive of this invention. Typically, the con-
centrates contain sufficient diluent to make them easy to
handle during shipping and storage. Suitable diluents for
the concentrates include any inert diluent, preferably an
20 oil of lubricating viscosity, so that the concentrate may
be readily mixed with lubricating oils to prepare lubri-
cating oil compositions. Suitable lubricating oils which
can be used as diluents typically have viscosities in the
range from about 35 to about 500 Saybolt Universal Seconds
25 (SUS) at 100°F (38°C), although an oil of lubricating
viscosity may be used.

Other additives which may be present in the
formulation include rust inhibitors, foam inhibitors,
corrosion inhibitors, metal deactivators, pour point
30 depressants, antioxidants, and a variety of other
well-known additives.

The following examples are offered to speci-
fically illustrate the invention. These examples
and illustrations are not to be construed in any way as
35 limiting the scope of the invention.

EXAMPLESExample 1Preparation of C₁₄-C₁₈ Alkyl Catechol

To a 3-liter flask, equipped with stirrer, Dean
5 Stark trap, condensor and nitrogen inlet and outlet was
charged 759 gm. of a C₁₄-C₁₈ alpha-olefin (2% C₁₄;
30% C₁₅; 30% C₁₆; 28% C₁₇; and 10% C₁₈), 330 gms. of pyro-
catechol, 165 gms. of a sulfonic acid cation exchange
resin (polystyrene cross-linked with divinylbenzene) cata-
10 lyst (Amberlyst 15 supplied by Rohm and Haas) and 240 mls.
toluene. The reaction mixture was heated at 150°C to
160°C for about 7 hours with stirring under a nitrogen
atmosphere. The reaction mixture was stripped by heating
to 160°C under vacuum (0.4 mm Hg). The product was fil-
15 tered hot over super cell (SCC) to afford 908.5 gms. of
C₁₄-C₁₈ alkyl substituted pyrocatechol. The product had a
hydroxyl number of 259. In a similar manner, by substi-
tuting an equivalent amount of each of a C₁₂ alpha-olefin,
a C₁₄ alpha-olefin and a C₁₈ alpha-olefin in the above
20 procedure, the corresponding alkyl catechols are prepared.

Example 2Preparation of C₁₆-C₂₆ Alkyl Catechol

To a 3-liter flask, equipped with stirrer, Dean
Stark trap, condensor and nitrogen inlet and outlet was
25 added 759 gms. of a mixture of C₁₆ to C₂₆ olefin (less
than C₁₄ - 2.7%; C₁₄ - 0.3%; C₁₆ - 1.3%; C₁₈ - 8.0%; C₂₀ -
44.4%; C₂₂ - 29.3%; C₂₄ - 11.2%; C₂₆ - 2.2%; C₂₈ - 0.4%;
C₃₀ - 0.2%) (containing at least 40% branching (available
from Ethyl Corp.), 330 gms. of pyrocatechol, 165 gms. of a
30 sulfonic acid cation exchange resin (polystyrene cross-
linked with divinylbenzene) catalyst (Amberlyst 15® avail-
able from Rohm and Haas, Philadelphia, Pennsylvania) and
240 ml. toluene. The reaction mixture was heated to 150°C
to 160°C for about 7 hours with stirring under a nitrogen
35 atmosphere. The reaction mixture was stripped by heating
to 160°C under vacuum (0.4 mm Hg). The product was

1 filtered hot over diatomaceous earth to afford 971 gms. of
a liquid C₁₆ to C₂₆ alkyl-substituted pyrocatechol.

Example 3

Preparation of Borated C₁₄-C₁₈ Alkyl Catechol

5 To a 906 grams of C₁₄-C₁₈ alkyl catechol was
added 124 grams boric acid and 900 ml of toluene. The
reaction mixture was heated at 105 to 118°C for about 6
hours under a nitrogen atmosphere at azeotropic condi-
tions. 93 mls of water were collected by a Dean Stark
10 trap. The reaction product was filtered and stripped on a
roto evaporator under vacuum to 155°C to yield 930 grams
of the title product.

Example 4

An oil blend was prepared as indicated in
15 Table I using CitCon 100N oil and containing 1.0% by
weight of the borated alkyl catechol prepared according to
Example 3.

TABLE I

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<u>Formulation</u>	<u>Time - Days</u>	<u>Observation</u>
Base oil	1-3 clear	bright and
25 Base oil + 1% by weight Borated Alkyl Catechol of Example 3	1 precipitate formed	hazy and

Example 5

30 One part by weight of the borated alkyl catechol
prepared according to Example 3 and 3 parts by weight of a
48% by weight of polyisobutenyl succinimide (prepared by
reacting polyisobutenyl succinic anhydride wherein the
number average molecular weight of the polyisobutenyl was
about 950 and tetraethylenepentamine in a mole ratio of
35 amine to anhydride of 0.87) solution in oil (CitCon 100N)
were heated together with mixing on a hot plate at 100°C
for 0.5 hours.

1 20 ml of the reaction mixture was placed in a
100 ml beaker and stored. A 100 ml beaker containing
20 ml of only the borated alkyl catechol which had been
heated up to 150°C and no succinimide was also stored for
5 a comparison.

The borated alkyl catechol hydrolyzed and formed
a skin on its surface as it cooled down (approx. 1/4-hr).
The borated alkyl catechol-succinimide complexed material
remained bright and clear after one week in storage. Even
10 after three weeks, the sample remained clear.

Example 6

Tests were carried out which demonstrate the
improvements in fuel economy obtained by adding lubricat-
ing oil compositions of this invention to the crankcase of
15 an automobile engine.

In this test, a 350 CID Oldsmobile engine was
run on a dynamometer. An engine oiling system was devised
in order to provide proper lubrication to the engine and
also to provide the capability to change the oil without
20 stopping the engine. Basically a dry sump system was used
with an external pump providing lubrication to the engine.
This pump was connected through valves to four external
sumps. The positioning of the valves determined the oil
used.

25 This test was conducted with base oil and then
with the same oil containing 1% by weight of the borated
C₁₄-C₁₈ alkyl catechol prepared according to Example 3.
The percent improvements in fuel economy using the compo-
sitions of the invention as compared to the base oil is
30 shown in Table II.

TABLE II

Fuel Economy Over Baseline
Concentrations of Sample

35	Concentration	
	<u>% by weight</u>	<u>% Improvement</u>
	1	1.5

1 The comparisons described above were made with fully for-
mulated Exxon 150N oil containing 3.5% of a polyisobutenyl
succinimide of tetraethylenepentamine, 30 mmols/kg over-
based magnesium hydrocarbyl sulfonate, 20 mmols/kg of
5 overbased calcium hydrocarbyl sulfonate phenate,
8.5 mmols/kg zinc 0,0-di(2-ethylhexyl) dithiophosphate,
8 mmols/kg of a mixed zinc dialkyldithiophosphate from
sec-britanol, methylisobutyl carbinol, 0.5% sulfurized
calcium polypropylene phenate, 1.5% of a sulfurized molyb-
10 dic acid succinimide complex and sufficient amount of an
amine substituted ethylene/propylene copolymer to give a
10W30 oil in this formulation and improver.

Also, formulated crankcase oils each containing
0.5% to 2% by weight of borated C₁₈-C₂₄ monoalkyl cate-
15 chol, borated C₁₄-C₁₈ dialkyl catechol and the like in the
place of borated C₁₄-C₁₈ alkyl catechol of Example 3 in
the above formulations are also effective in reducing fuel
consumption in an internal combustion engine.

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1 CLAIMS:

1. A lubricating oil additive comprising a complex obtainable by reacting a borated alkyl catechol with an oil soluble alkyl or alkenyl mono or bis succinimide.

5 2. An additive as claimed in Claim 1, wherein the alkyl group of said borated alkyl catechol contains from 10 to 30 carbon atoms and said succinimide is a polyisobutenyl succinimide of a polyalkylene polyamine.

3. An additive as claimed in Claim 2, wherein said
10 alkyl group of said borated alkyl catechol is a mixture of alkyl groups containing from 14 to 26 carbon atoms.

4. An additive as claimed in Claim 2 or 3, wherein said succinimide is a polyisobutenyl succinimide of triethylenetetramine or a polyisobutenyl succinimide of
15 tetraethylenepentamine.

5. A lubricating oil composition comprising an oil of lubricating viscosity and, in an amount effective to reduce friction, an additive as claimed in any one of Claims 1 to 4.

20 6. A method of reducing the fuel consumption of an internal combustion engine comprising treating the moving surfaces thereof with a lubricating oil composition as claimed in Claim 5.

7. A lubricating oil composition comprising an oil
25 of lubricating viscosity and 0.1 to 4 weight percent of a borated alkyl catechol and an effective amount of an alkenyl mono or bis succinimide to stabilize the borated alkyl catechol against hydrolysis.

8. A lubricating oil composition according to Claim
30 7, wherein the alkyl group of said borated alkyl catechol contains from 10 to 30 carbon atoms and said alkenyl succinimide is a polyisobutenyl succinimide of a polyalkylene polyamine.

9. A lubricating oil composition according to Claim 8,
35 wherein said alkyl group of said borated alkyl catechol is a mixture of alkyl groups containing from 14 to 18 carbon atoms.

- 1 10. A lubricating oil composition according to
Claim 8 or 9, wherein said alkenyl succinimide is a
polyisobutenyl succinimide of triethylenetetramine or a
polyisobutenyl succinimide of tetraethylenepentamine.
- 5 11. A lubricating oil composition according to
Claim 8, wherein the alkyl group of said borated alkyl
catechol is a mixture of alkyl groups containing from
14 to 26 carbon atoms.
- 10 12. A method of reducing the fuel consumption of an
internal combustion engine comprising treating the moving
surfaces thereof with a complex as claimed in Claim 1.
13. A lubricating oil composition comprising
(a) an oil of lubricating viscosity; and
(b) an effective amount of each of the following:
- 15 1. an alkenyl succinimide,
2. a borated alkyl catechol friction modifier,
3. a Group II metal salt of a dihydrocarbyl
dithiophosphoric acid,
4. a neutral or overbased alkali or alkaline
20 earth metal hydrocarbyl sulfonate or mixture there-
of, and
5. a neutral or overbased alkali or alkaline
earth metal alkylated phenate, or mixture thereof.
- 25 14. A lubricating oil composition according to
Claim 13, wherein
(1) said alkenyl succinimide is a polyisobutenyl
succinimide of a polyalkylene polyamine;
(2) said borated alkyl catechol is a borated C₁₄-C₁₈
alkyl catechol;
- 30 (3) said metal salt of the dihydrocarbyl dithiophos-
phoric acid is zinc dialkyl dithiophosphate wherein the
alkyl group contains from 4 to 12 carbon atoms;
(4) said metal of the neutral or overbased alkali or
alkaline earth metal sulfonate is calcium, magnesium or
35 barium or a mixture thereof; and
(5) said metal of the neutral or overbased alkali or
alkaline earth metal phenate is calcium, magnesium or
barium.

1 15. A lubricating oil composition according to
Claim 14, wherein

(1) said alkenyl succinimide is a polyisobutenyl
succinimide of triethylenetetramine or a polyisobutenyl
5 succinimide of tetraethylenepentamine;

(2) said borated alkyl catechol is a borated C₁₄-C₁₈
alkyl catechol;

(3) said metal salt of the dihydrocarbyl dithiophos-
phoric acid is zinc 0,0-di(2-ethylhexyl)dithiophosphate,
10 zinc 0,0-di(isobutyl/mixed primary hexyl)dithiophosphate, .
or zinc 0,0-di(sec-butyl/mixed secondary hexyl)dithiophos-
phate;

(4) said metal salt of the sulfonate is an overbased
magnesium or calcium hydrocarbyl sulfonate; and

15 (5) said metal salt of the phenate is an overbased
sulfurized calcium or magnesium monoalkylated phenate.

16. A method of reducing the fuel consumption of an
internal combustion engine comprising treating the moving
20 surfaces thereof with a composition according to Claim 13,
14 or 15.

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