2,805,998

LUBRICANT COMPOSITIONS

Troy L. Cantrell, Lansdowne, Pa., and John G. Peters, Audubon, N. J., assignors to Gulf Oil Corporation, Pittsburgh, Pa., a corporation of Pennsylvania

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This invention relates to improved lubricants for use in internal combustion engines and in particular to lubricating compositions having improved detergent characteristics.

In lubricating internal combustion engines, such as automotive and aviation engines, lubricants such as mineral lubricating oils frequently prove unsatisfactory because of their tendency to deposit varnish, gum and sludge on the metallic engine surfaces coming in contact with the lubricants. The formation of these undesirable deposits is accelerated when severe operating conditions are encountered. Because of the current trend toward higher efficiency or higher output per unit weight of engine, the lubricant has been subjected to accelerated deteriorating influences. As a result of increased operating loads placed on lubricants, some mineral oils containing oil deteriorating inhibitors have been unsatisfactory because the inhibitor itself broke down.

Various metallic salts have been incorporated in mineral lubricating oils to improve the detergent characteristics of such oils. While the metallic salts have given improved detergent properties, their use has not met with complete satisfaction, particularly in aviation engines. Metallic salts are disadvantageous in that they form an ash when burned, thus increasing the possibility of engine failure by reason of ash deposition.

We have found that a lubricating composition to which a relatively small amount of an aromatic hydroxy alkyl amine has been added substantially overcomes the accumulation of harmful engine deposits. For instance, we have found that the presence of a relatively small amount of an aromatic hydroxy alkyl amine in a mineral lubricating oil substantially inhibits the formation of engine deposits normally encountered when using mineral lubricating oils. The aromatic hydroxy alkyl amine which is used in accordance with our invention has the further advantage in that it forms substantially no ash upon being burned. The combustion chambers of an engine and particularly the pistons of an engine lubricated with a mineral lubricating oil containing an aromatic hydroxy alkyl amine are substantially cleaner than engines lubricated with the mineral lubricating oil alone.

The aromatic hydroxy alkyl amine which can be incorporated in the lubricating compositions in accordance with our invention is advantageously prepared by reacting an alkylene oxide with a primary aromatic amine of the benzene series. The alkylene oxide which can be used in accordance with the present invention is advantageously one having from 2 to 4 carbon atoms such as ethylene oxide, propylene oxide, and butylene oxide. The primary aromatic amine which can be used is advantageously aniline and its mono- and di-methyl derivaties, i. e., toluidine and xylidine. While we can employ a single xylidine in the synthesis of the aromatic hydroxy alkyl amine, for reasons of economy we advantageously employ commercially available mixtures of the isomeric xylidines. The product obtained when a mixture of isomeric xylidines is used in a mixture of the isomeric xylyl alkanol amines. Specific examples of the primary aromatic amines which we can use are aniline, o-toluidine, m-toluidine, p-toluidine, 2,3-xylidine, 2,4-xylidine, 2,5-xylidine, 2,6-xylidine, 3,4-xylidine and 3,5-xylidine. The aromatic hydroxy alkyl amine is generally employed in an amount between about 0.5 and 20.0 percent by weight based on the weight of the oil. In some instances, excellent results can be obtained with about 1.0 to about 10.0 percent by weight of the aromatic hydroxy alkyl amine.

Examples of the aromatic hydroxy alkyl amines which we can use are phenyl diethanolamine, phenyl dipropanolamine, phenyl dibutanolamine, m-tolyl diethanolamine, m-tolyl dipropanolamine, m-tolyl dibutanolamine, o-tolyl diethanolamine, o-tolyl dipropanolamine, o-tolyl dibutanolamine, p-tolyl diethanolamine, p-tolyl dipropanolamine, p-tolyl dibutanolamine, 2,3-xylyl diethanolamine, 2,3-xylyl dipropanolamine, 2,3-xylyl dibutanolamine, 2,4-xylyl diethanolamine, 2,4-xylyl dipropanolamine, 2,4-xylyl dibutanolamine, 2,5-xylyl diethanolamine, 2,5-xylyl dipropanolamine, 2,5-xylyl dibutanolamine, 2,6-xylyl diethanolamine, 2,6-xylyl dipropanolamine, 2,6-xylyl dibutanolamine, 3,4-xylyl diethanolamine, 3,4-xylyl dipropanolamine, 3,4-xylyl dibutanolamine, 3,5-xylyl diethanolamine, 3,5-xylyl dipropanolamine, 3,5-xylyl dibutanolamine, and mixtures thereof.

The aromatic hydroxy alkyl amines can be prepared by any of the methods known in the art. The reaction of a primary aromatic amine with an alkylene oxide is highly exothermic and for this reason efficient means for removing heat during the reaction period must be established. One method of producing an aromatic hydroxy alkyl amine is described in U. S. Patent No. 2,432,023 which issued on December 2, 1947, to Lecher et al. In accordance with the patent, the reaction is controlled by adding a small amount of the expected reaction product along with the reactants. For example, in producing phenyl diethanolamine from aniline and ethylene oxide the reaction is advantageously carried out in the presence of at least 10 percent by weight, based on the weight of the primary amine, of phenyl diethanolamine. The reaction proceeds smoothly at temperatures up to about 300° F. The amounts of reactants employed can be varied. However, we prefer to employ the alkylene oxide and the aromatic amine in a molecular ratio of about 2 to 1. For example, in reacting propylene oxide with a xylidine or a mixture of the isomeric xylidines, we advantageously employ 2 moles of propylene oxide with 1 mole of the xylidine.

The following example will illustrate the preparation of a mixture of xylyl dipropanolamines which can be used in a lubricating oil to improve its detergency. The xylidines used in preparing the product are a commercially available mixture of xylidines. The mixture of xylidines has a specific gravity of from 0.97 to 0.99 and a boiling point of from 213° C. to 226° C. The mixture is liquid at room temperature and is insoluble in water but soluble in alcohol and ether.

Example 1

In a stainless steel reaction vessel equipped with a reflux condenser there were added 1694 grams (14 moles) of a commercial mixture of xylidines having the above properties. The reaction vessel was placed in a larger vessel filled with water by means of which the reaction temperature could be controlled. Thereafter, 1604 grams (28 moles) of propylene oxide were slowly added to the reaction vessel. The contents of the reaction vessel were then agitated for 3 hours at room temperature after which the reaction product was heated to 280°

F. to remove water. The dehydrated reaction product after filtration had the following properties.

Gravity, ° API	5.9
Viscosity, SUS at 210° F.	52.8
Color (NPA)	5.25
Neutralization number	0.04
Ash, as sulfate percent	0.002

In order to illustrate the detergent properties of a mineral lubricating oil containing the product obtained in accordance with Example 1, an aviation oil was employed. The physical characteristics of the aviation oil and the same oil containing 1 percent by weight of the product of Example 1 were as follows:

	Aviation Oil	Aviation Oil Containing 1% by Weight of Product of Example 1
Gravity, ° API	26.0	25.8
Viscosity: SUS at 210° F.	121.1	117.7
Flash Point, ° F.	535	535
Fire Point, ° F.	600	600
Pour Point, ° F.	0	−10
Color (NPA)	4.75	4.75
Carbon Residue, Percent	0.51	0.55
Ash, Percent	trace	trace

The aviation oil and the aviation oil containing the product of Example 1 were tested in accordance with the following test procedure. In this test a single cylinder Waukesha L head engine developing about 4.4 horsepower is used. The engine, which is equipped with an aluminum piston, is charged with about 6.6 pounds of test oil and operated at full load at a speed of about 1200 revolutions per minute with a coolant temperature of about 350° F. and an oil temperature of about 185° to about 195° F. At the end of 50 hours of operation, or sooner if engine failure is encountered, the engine is disassembled and inspected for deposits and various parts are rated on a cleanliness scale of 0 to 10. A rating of 0 is assigned to parts which are free of deposits and a rating of 10 to parts on which deposits have reached a maximum or caused engine failure.

When the aviation oil designated above was subjected to the above test procedure, engine failure occurred in 32 hours because of excessive carbon deposits. When the aviation oil containing 1 percent of the reaction product of Example 1 was similarly tested an engine rating of 1 was obtained. In the latter test there were no rings stuck and no heavy hard deposits.

Example 2

The procedure outlined in Example 1 was repeated with the exception that 12 moles of propylene oxide were reacted with 6 moles of aniline. The reaction was carried out in the presence of a Filtrol catalyst which was removed after completion of the reaction by filtration. The reaction product consisting predominantly of phenyl dipropanolamine had the following properties:

Gravity, ° API	0.9
Viscosity, SUS at 210° F.	93.0
Color ASTM Union	3.25

One percent by weight of the phenyl dipropanolamine of Example 2 was then added to the aviation base oil.

The oil was then run in the Waukesha engine for 50 hours. When the engine was disassembled, there were no rings stuck and no heavy hard deposits. The engine was given a rating of 1. The effectiveness of the aromatic hydroxy alkyl amines in preventing the formation of engine deposits is thus readily apparent.

In preparing the improved lubricants of our invention we can also incorporate in the lubricants other addition agents normally added to lubricating oils for a specific purpose such as anti-oxidants, pour point depressants, corrosion inhibitors, foam suppressants, viscosity index improvers, and the like, without adversely affecting the detergent benefits derived by the composition of this invention.

While we have demonstrated the improved detergency properties with respect to mineral lubricating oils, it is to be understood that the invention encompasses the use of other hydrocarbon lubricating oils, such as those produced in the Fischer-Tropsch and related processes.

While our invention is described above with reference to various specific examples and embodiments, it will be understood that the invention is not limited to such examples and embodiments and may be variously practiced within the scope of the claims hereinafter made.

We claim:

1. A lubricating composition comprising a major amount of a lubricating oil and a minor amount, sufficient to improve the detergent characteristics of said lubricating oil, of an aromatic dialkanol amine wherein the aromatic radical is selected from the group consisting of phenyl, tolyl and xylyl, and the alkanol radicals each contain from 2 to 4 carbon atoms, said minor amount being from about 0.5 to about 10 percent by weight of the total composition.

2. A lubricating composition comprising a major amount of a lubricating oil and a minor amount, sufficient to improve the detergent characteristics of said lubricating oil, of a phenyl dialkanol amine wherein each alkanol radical contains from 2 to 4 carbon atoms, said minor amount being from about 0.5 to about 10 percent by weight of the total composition.

3. A lubricating composition comprising a major amount of a lubricating oil and a minor amount, sufficient to improve the detergent characteristics of said lubricating oil, of phenyl dipropanol amine, said minor amount being about 1 percent by weight of the total composition.

4. A lubricating composition comprising a major amount of a lubricating oil and a minor amount, sufficient to improve the detergent characteristics of said lubricating oil, of tolyl dipropanol amine, said minor amount being about 1 percent by weight of the total composition.

5. A lubricating composition comprising a major amount of a lubricating oil and a minor amount, sufficient to improve the detergent characteristics of said lubricating oil, of xylyl dipropanol amine, said minor amount being about 1 percent by weight of the total composition.

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