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LUBRICATING OIL

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Our invention relates to an improved lubricating oil and more particularly to a lubricating oil having a low cold test and a high film strength.

In the production of high grade lubricating oils for present-day motor equipment certain characteristics must be obtained in the finished lubricant other than viscosity, flash point, freedom from grit and other abrasive substances which were previously the controlling factors. Present-day high grade lubricants must have, in addition to the above low cold test or solidification points, high film strength and increased oiliness or unctuosity.

Low cold test or solidification point of a lubricant is obtainable in the present-day art by the very costly de-waxing processes. In addition to the very high cost of original equipment for de-waxing and the continued high cost of operation of de-waxing equipment, there is another objection to this method of obtaining low cold test due to certain objectionable changes in the nature of the lubricant as a direct result of the removal of the wax.

Another method of lowering the cold test of a lubricant has been described in United States Letters Patent 1,815,022, granted July 14, 1931, to Garland H. B. Davis.

In the Davis invention, a synthetic hydrocarbon oil is formed by condensing a wax hydrocarbon material with an aromatic hydrocarbon or by condensing a halogenated paraffin with an aromatic hydrocarbon. This synthetic oil, when added to a lubricating oil containing a solid wax component, will reduce the pour-point thereof. The film strength of the lubricant, however, is not improved and may even be decreased.

One object of our invention is to provide a lubricant having a lowered cold test and an increased film strength.

Other objects of our invention will appear from the following description:

In general, our method consists of the condensation of aliphatic or open chain compounds, preferably hydrocarbons, with aromatic compounds and particularly aromatic hydrocarbons by the reaction known as "Friedel-Crafts reaction," for example, in order to produce condensation products of such aliphatic and aromatic compounds. The thus prepared condensation product is then treated with a sulphur halide such as sulphur chloride to form a lubricating oil addition agent containing sulphur and halogen. The purified treated condensation product is added to lubricants in small amounts and optionally in a blend with small amounts of halogenated fatty acids.

The following examples of constituents suitable for use in the production of the addition agent of this invention are given for purposes of illustration only and are not intended in any way to limit the scope of the invention. Suitable material for use as open chain compounds are:

Paraffin wax.
Wax distillates.
Foots oil.
Intermediates obtained in processing crude wax and wax distillate.
Wax fractions.
High boiling fractions from crude oil.
Long chain hydrocarbons.
Organic compounds in which the major proportion of the carbon in the molecule is in open chain combination, e. g.:
- Petroleum naphthenates with long chain substituents on the naphthenic nucleus.
- Olefins made from petroleum wax.
- Oleic acid.
- Methyl oleate.
- Oleyl alcohol.

Naturally occurring waxes, e. g.:
- Palm wax.
- Beeswax.
- Carnauba wax.
- Candelilla wax.
- Spermaceti.

Long chain esters, e. g.:
- Methyl stearate.
- Ethyl oleate.
- Cetyl acetate.
- Oleyl butyrate.

Long chain alcohols, e. g.:
- Lauryl.
- Oleyl.
- Cetyl alcohols.

Aromatic compounds which may be condensed with the above open chain compounds include:

Benzene.
Toluene.
Naphthalene.
Butyl naphthalene.
Anthracene.
Diphenyl.
Diphenyl oxide.
Diphenylene oxide.
Diphenyl sulphide.
Diphenyl ethylene.
Mixtures rich in aromatic compounds such as coal tar fractions, e. g.:
- Extracts from solvent refining lubricating oil.

The aliphatic compound may be halogenated and then condensed with the aromatic compound by the "Friedel-Crafts reaction" or one or both compounds may be unsaturated and condensation take place at the double bond. Chemical unsaturation in the condensation product is advantageous in some cases.

For example, wax is chlorinated by direct chlorination until it contains about the calculat-

ed amount of halogen for a mono-halogen product. This will vary rather widely with the approximate average molecular weight of the wax being halogenated and may range from 10% or less of chlorine on up to 20% or more. The temperature is controlled during the halogenation step to avoid temperatures much above 300° F. Aromatic hydrocarbons such as naphthalene or anthracene are then mixed with approximately an equal weight of aluminum chloride and the chlorinated oil added slowly, while maintaining a temperature not to exceed about 160° F. An inert solvent such as carbon disulfide, petroleum ether, ether, acetone etc. may be used for the reacting mixture which should be constantly agitated until the reaction is completed, which is apparent from the lack of formation of large volumes of hydrogen chloride gas when fresh chlorinated wax is added. This also controls the proportions in which the chlorinated wax is added. When the reaction is apparently complete, it is desirable to increase the temperature of the reacting mixture to expel any remaining hydrogen chloride. The mass is allowed to stand until the aluminous sludge settles out or the sludge may be removed by mechanical means such as filtration. The reaction product is then placed in distilling equipment and the solvent and unreacted wax removed with as little heat as possible and preferably under a high vacuum. The recovered condensation product is a liquid organic mixture containing slight traces of halogen, usually less than 0.4%.

The above condensation product may then be treated with sulphur chloride. Sulphur chloride may be added to the original condensation product direct by bringing the reacting mixture to a temperature of from 200° F. to 300° F. The condensation product may be blended with 10% sulphur chloride or with 10% sulphur-chloride in the presence of a small amount of hydrated lime and heated to about 200° F. to 300° F. and constantly stirred until the reaction is complete. The reaction mixture, in some cases, should be held at a high temperature for some time, say six hours or more, even though the reaction is apparently complete after 30 minutes or less. If lime is used in the reaction mixture, the mixture is then filtered to remove the lime and/or calcium salts. Or a sufficient quantity of lime may be added to the reaction product to neutralize any free inorganic acidity and upon filtering the product is then ready for use. Other means of purification, such as contacting with clay, use of solvents and vacuum distillation, may be employed but the above described simple method is satisfactory. The condensation product may be treated with 5% or less, 10%, 15%, 20% of sulphur chloride, or more.

As an example of the cold test lowering of the above products, a sample of high quality 350 viscosity steam refined stock with a 22° F. cold test was blended with a sulphur chloride treated condensation product with the following results:

	Cold test, 0° F.
Original 350 vis. lubricant	22
99.5% original lubricant plus 0.5% condensation product treated with 10% sulphur chloride	−20
99.5% original lubricant plus 0.5% condensation product treated with 20% sulphur chloride	−4

From the above table the efficiency of our product in lowering cold test is fully demonstrated. In the tests above, only 0.5% of the product of our invention was added to the lubricant, but with poorer quality lubricants and/or with higher cold test lubricants larger quantities may be used and may be as much as 10% or more of the volume of the original lubricant. In the above lubricant 0.25% of the condensation product treated with 10% sulphur chloride gave a cold test of 2° F. In the same lubricant 0.1% of the condensation product treated with 10% sulphur chloride in 99.9% of the lubricant gave a cold test of 19° F. Therefore, the minimum amount in the above lubricant is about 0.1% by volume.

To prove the increased film strength of lubricants prepared according to our invention, we employ the new type Timken testing machine manufactured by the Timken Roller Bearing Company. In this machine lubricants are used between two rubbing surfaces under increasing pressure or load until the lubricant breaks down and allows metal seizure. The load required to cause seizure is a measure of the film strength of the lubricant under test. These loads converted into pounds per square inch on the rubbing surfaces gives a good comparison of the film strength of any two lubricants. In the following examples a high quality S. A. E. 40 lubricant was used and the results are reported in pound weights added to obtain film rupture and these pound weights converted into approximate pounds per square inch.

	Oil tested	Pound weights	Approximate pounds per square inch
1	Original S. A. E. 40 mineral oil	11.3	7,000
2	99% S. A. E. 40 lubricant plus 1% of condensation product	10.3	6,300
3	99.5% S. A. E. 40 lubricant plus 0.5% of condensation product treated with 10% sulphur chloride	18.05	11,700

From the above it is noted that the addition of the condensation product direct to the lubricant lowers the film strength of the original lubricant. It also shows that if the condensation product is treated with sulphur chloride, the film strength of the original lubricant is greatly improved.

To 98.5% of another sample of the S. A. E. 40 lubricant mentioned above, we added 1% of the condensation product treated with 10% sulphur chloride and ½% chlorinated stearic acid prepared by passing free chlorine into stearic acid. The original S. A. E. 40 lubricant had a cold test of 22° F.; the blended product had a cold test of −18° F. The original lubricant had a film strength of 7,000 pounds per square inch (11.3 pound weight); the blended lubricant had a film strength of 31,250 pounds per square inch (55.45 pound weight). The original lubricant had a coefficient of friction of 0.135 to 0.140 when tested on the Herschel friction testing machine; the blended lubricant had a coefficient of friction of 0.075 to 0.080.

Small amounts of chlor stearic or other halogenated organic acids in any of the mixtures described in this invention will improve the film strength and decrease the coefficient of friction (give greater oiliness). 0.5% chlor stearic acid in the S. A. E. 40 lubricant gives a film strength of only 27,500 pounds per square inch (48.05 pound weight); therefore, the combination of chlorinated stearic acid and sulphur chloride-treated condensation products give higher film strength than either alone will give.

Our addends are admirably adapted for use in

oil of lubricating viscosity of all types including those designated for use in automotive crankcases, Diesel oils, hydrogenated, polymerized and otherwise synthetically treated oils such as voltolized oils, aluminum chloride treated oils, and the like. Furthermore the lubricating oil may consist in whole or in part of shale oil, animal or vegetable oils such as castor oil, lard oil, corn oil, cottonseed oil, and the like, and any other oils of lubricating viscosity. Furthermore, our addends are advantageously blended in gasoline and other petroleum fuels either directly or after being blended first in a lubricating oil and then added to the fuel. Soap-thickened mineral oils of all types ranging from those showing only a slight increase in viscosity over that of the mineral oil alone to the semi-solid and solid greases containing fifty per cent or more of soap are amenable to treatment according to our invention. In making these greases, the usual soaps such as sodium stearate, aluminum stearate, calcium soaps of beta fat, and the like may be used to form the large part of the necessary soap. Various other thickening ingredients or materials for other purposes may be added. These include yarn, hair, graphite, glycerol, water, lampblack, mica, zinc dust, litharge, and the like.

Other modes of applying the principle of our invention may be employed instead of the one explained, change being made as regards the materials employed, provided the ingredients stated by any of the following claims or the equivalent of such stated ingredients be employed.

We, therefore, particularly point out and distinctly claim as our invention:

1. An improved lubricating oil comprising a major proportion of mineral oil and a minor amount of a sulphur-chloride treated condensation product of a halogenated paraffin and an aromatic hydrocarbon.

2. An improved lubricating oil comprising a major proportion of mineral oil and a minor amount of a sulphur-chloride treated condensation product of a halogenated wax and an aromatic hydrocarbon.

3. An improved lubricating oil comprising a major proportion of mineral oil and a minor amount of a sulphur-chloride treated condensation product of a halogenated paraffin wax and an aromatic hydrocarbon.

4. An improved lubricating oil comprising a major proportion of lubricating oil and from about 0.1% to about 10% by volume, based on the original oil, of a sulphur-chloride treated condensation product of a halogenated paraffin and an aromatic hydrocarbon.

5. An improved lubricating oil comprising a major proportion of lubricating oil and from about 0.1% to about 10% by volume, based on the original oil, of a sulphur-chloride treated condensation product of halogenated paraffin wax and an aromatic hydrocarbon.

6. An improved lubrication oil comprising a major proportion of mineral oil and a minor amount of a sulphur-chloride treated condensation product of an aliphatic hydrocarbon and an aromatic compound.

7. An improved lubricating oil comprising a major proportion of mineral oil and a minor amount of a sulphur-chloride treated condensation product of an unsaturated aliphatic hydrocarbon and an aromatic compound.

8. An improved lubricating oil comprising a major proportion of mineral oil and a minor amount of a sulphur-chloride treated condensation product of an aliphatic hydrocarbon and an unsaturated aromatic compound.

9. An improved lubricating oil comprising a major proportion of mineral oil and a minor amount of a sulphur-chloride treated condensation product of an unsaturated aliphatic compound and an aromatic compound.

10. An improved lubricating oil comprising a major proportion of mineral oil and a minor amount of a sulphur-chloride treated condensation product of a halogenated aliphatic compound and an aromatic compound.

11. An improved lubricating oil comprising a major proportion of mineral oil and a minor amount of a sulphur-chloride treated condensation product of an aliphatic compound and an aromatic compound.

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