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HIGH PRESSURE LUBRICANT

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The present invention relates to lubricants, especially to the class known as extreme pressure lubricants and more particularly to improved lubricants of this type and the method of manufacturing same.

The invention will be fully understood from the following description:

Within the last few years a considerable demand has arisen for lubricants, both solid and liquid, which are capable of withstanding loads which are in considerable excess of those possible with straight or unblended hydrocarbon oils. This is especially the case for automobile transmissions and other power transferring mechanisms in which hypoid and other type gears are used.

Among the compounds that have been proposed are those containing sulfur and chlorine in combination. These are fully capable of withstanding the pressures and otherwise give good lubricating characteristics but there are objections which have limited if not prevented general use. The main objection is that the compounds as ordinarily prepared are corrosive and since it is impossible to avoid small quantities of moisture, this results in discoloration of the gears and other mechanism, and in time, corrosion, pitting and rusting. Further, as a result of this corrosive action, there are formed oil insoluble abrasive materials which materially lower the lubricating value of such blends.

In the present invention a compound of the sulfur-chlorine type is proposed which while carrying the load only corrodes or rusts steel surfaces to a limited degree and then for only a period of time which is short enough to be tolerated. Furthermore the present compound is effective over substantially the whole life of the oil, while in the case of many prior compounds the valuable load-carrying properties are lost after a relatively short period.

In the present high pressure compound use is made of a hydrocarbon base oil which is selected for the particular use to which the compound is to be adapted. Its viscosity is ordinarily above 100 seconds Saybolt at 210° F. and may be in excess of 200 or even 300, depending on the particular service. The oil may be derived from a naphthenic or a paraffinic crude and the cut may be a distillate or residue as desired. The flash point is ordinarily not of much significance for transmission oils but it is usually above 400° F. or 450 or even higher. The present compounds may be liquid or they may be solidified in which case aluminum, soda, or lime soaps such as those derived from tallow, stearic acid and the like may be added in proportions ranging from 10 to 50%.

The sulfur-bearing compound is of the type described as "corrosive" which means that it discolors a bright copper strip immersed therein but it in fact has no appreciable action on steel gears or metal containers in which it is kept. The amount of corrosive sulfur should be in excess of .2% and ordinarily from .30 to 3% or thereabout. Various compounds of sulfur may be used but it should be understood that sulfur exists in "non-corrosive" as well as "corrosive" forms and therefore it is desirable to determine the amount of "corrosive" sulfur and to blend on this basis. "Corrosive" sulfur may be determined as the difference between total sulfur in two samples, one of which has been deprived of "corrosive" sulfur by digesting the same with a finely divided metal, for example copper, over a period of several hours at steam bath temperature. Its sulfur content may be determined by any standard method, for example by the method devised by the American Society for Testing Materials, D—129—27, described in report of Committee D—2 on Petroleum Products and Lubricants and Method of Tests Relating to Petroleum Products, page 174, 1928.

As to the sulfur-containing compounds, fractions from certain crudes known to contain "corrosive" sulfur may be used and as an example of these residues from West Texas may be given, it being understood that other known crudes of the type may be used. Elemental sulfur may be added, alone or to fortify the other corrosive sulfur present. Animal, vegetable and fish oils may be sulfurized by treatment with raw or elemental sulfur at elevated temperatures. Organic polysulfides may also be used. For example, they may be prepared by chlorination of a mineral oil or wax by bubbling chlorine therethrough for a period of several hours or until it contains from 5 to 30% chlorine. The chlorine may then be substituted by sulfur through the reaction with alkaline polysulfides, for example sodium or potassium polysulfides. In this way chlorine may be entirely replaced from the compound. It is important that the chlorine compound should be added separately or in other words, that both the chlorine and sulfur be present in different compounds. This is important for it has been found that chlorine-sulfur compounds, for example those produced by treating fatty oils with sulfuryl chloride,

gradually lose chlorine on use and when chlorine is lost the sulfur is unable to carry the extreme pressure loads.

The chlorine compounds that can be used include chlorinated fatty oils and acids, chlorinated mineral oils and waxes such as paraffin wax, carbon tetra chloride and the like. It will be understood that fluorine and bromine compounds are equivalents for the chlorine compounds and can be substituted therefor although the chlorine compounds are to be preferred. The halogen atom is preferably attached to an aliphatic carbon atom, but in many instances chlorine attached to aromatic rings is satisfactory.

It has been found that relatively small amounts of the chlorine compound are effective, for example, as little as .20 to .60% although somewhat more can be used. In the proportion used the oil is not excessively corrosive even when first added in respect to the metal containers and machine parts, and it does not show rust or produce abrasive reaction products. The exact role of the chlorine is not understood but it is believed to be in the nature of an activator and in some manner permits the sulfur to "take hold" gradually so that the friction-load curve is smooth and without sharp breaks. Whatever the action may be its effect is permanent in spite of the gradual loss of chlorine; for example it has been found that in automobile transmissions after 100 or 200 miles the chlorine content has greatly diminished and after 400 to 500 miles it has completely disappeared. This corresponds to a running-in period of from 10 to 15 hours use. The sulfur content is intact and it has remained in a "corrosive" form. The oil, even after loss of chlorine is capable of withstanding high bearing and tooth pressures.

While not wishing to be limited to any particular theory of the roles of the two principal ingredients, it is believed that the chlorine serves to bring about reaction between the sulfur compound and the steel gear teeth and that once this has occurred to an extent sufficient to properly activate a substantial portion of tooth area, no additional chlorine is required, the remaining corrosive sulfur gradually completing the reaction after elimination of the chlorine.

It will be seen from the above that the present compound is practically non-corrosive to steel surfaces in the sense that it does not cause an appreciable rusting or formation of other abrasive compounds and furthermore that the chlorine is in sufficiently small quantities so that it may be eliminated altogether in from 10 to 15 hours of use. By an effective quantity it is intended to designate an amount sufficient to activate the sulfur and enable it to carry the loads at relatively low temperatures and without excessive friction.

The following examples may be considered to illustrate the composition and properties of suitable compounds made in accordance with the present invention.

Example I

The base oil used in all of the following compounds is as follows:

Gravity	degrees A. P. I.	26.4
Viscosity at 210° F	sec. Saybolt	120
Viscosity at 100° F	do	1565
Pour °F	degrees Fahrenheit	5
Flash	do	475
Color	R	½

To separate samples of the above base stock are added:

Sample 1, 8% of sulfurized pine oil made by cooking such oil with sulfur at a temperature of 425° F. and thereafter containing 4.0% "corrosive" sulfur.

Sample 2, 8% of sulfurized pin oil and 1½% of chlorinated paraffin wax containing 40% chlorine.

Sample 3, the base stock alone.

The following tests were made on the Almen machine as described in Automotive Industries vol. 67—#21 p. 650. The total number of weights carried in the tests, the weight at which friction increases suddenly (if such be the case), the final friction, temperature and condition of bearing are recorded for each test:

Sample	Wt. carried	Final friction	Sudden increase	Final temp.	Bearing condition
				°F.	
1	15	65	6 wts.	250	Deeply scratched.
2	15	30	None.	140	Excellent.
3	5	10		90	Seizure.

Example II

As a second example the following tests were run on the Almen machine. The base oil is a 100 sec. (Saybolt at 210° F.) Pennsylvania fraction. Various different solutions are added thereto in order to obtain the amounts of corrosive sulfur and chlorine as indicated. The number of weights carried is recorded, together with final friction, and comments as to bearing conditions. From an inspection of the table it will be seen that the best conditions are .2 to .5% or more corrosive sulfur with .3 to .5% chlorine. All except one of the samples within this range carried the full 15 weights and the other one carried 14 weights. The oil alone fails at about 5 or 6 weights. None of the oils are corrosive toward the metal gears and do not produce appreciable quantities of solid abrasive solvents.

Blend No.	Percent corrosive sulfur	Percent chlorine	Almen test		
			Weights carried	Final friction	Bearing condition
1	0.2	0.1	6	15	Welded.
2	0.2	0.3	15	29	Good.
3	0.2	0.5	14	28	Welded.
4	0.5	0.1	15	57	Scratched.
5	0.5	0.3	15	63	Scratched.
6	0.5	0.5	15	35	Good.
7	1.0	0.1	15	55	Scratched (poor).
8	1.0	0.3	15	48	Scratched.
9	1.0	0.5	15	53	Scratched.

Example III

As an illustration of the effect of chlorine elimination during use, the samples made up according to the present invention and samples of ordinary lubricants containing a "so called" extreme pressure agent produced by action of sulfur chloride on a fatty oil were subjected to treatment with steel wool at 300° F. for different periods of time. As indicated this treatment eliminated chlorine just as it is removed during use. Tests on the treated oils were then run on the Almen machine showing that the sulfur chloride oil rapidly lost its extreme pressure char-

acteristics while the oil produced according to the present invention did not.

Blend	300 °F. with 5% steel wool, hrs. of heating	Analysis of oil after treatment		Almen machine test	
		Percent sulfur	Percent chlorine	Load carried	Friction
Commercial sulfur chlorine lub	0	.99	.62	15	33
	6	1.11	.49	14	90
	8	1.15	.34	6	46
Example blend	0	.4	.5	15	30
	10	.4	Trace	15	60

In similar tests other samples of the oil are used in automobile transmission for a period of several days. Through the entire period they give satisfactory service and at the end thereof when the transmission is taken down, the oil samples are found to contain substantially the initial amount of corrosive sulfur and to be free of chlorine. Metal parts are not appreciably corroded and there is no evidence of rusting.

The present invention is not to be limited to any theory of the mechanism of extreme pressure lubricants of the present type, the effect of sulfur and chlorine, nor to any particular sulfur or chlorine containing compound, but only to the following claims in which it is desired to claim all novelty inherent in the invention.

We claim:

1. An improved lubricant for extreme pressure conditions comprising a major proportion of a mineral lubricating oil, a minor proportion of halogenated petroleum wax and an agent containing "corrosive" sulfur.

2. An improved lubricant for extreme pressure conditions comprising a major proportion of mineral lubricating oil and minor proportions of chlorinated petroleum wax and an agent containing "corrosive" sulfur selected from the group consisting of elemental sulfur, and sulfurized animal, vegetable, marine and mineral oils.

3. An improved lubricant for extreme pressure conditions comprising a major proportion of mineral lubricating oil and minor amounts of chlorinated paraffin wax and "corrosive" sulfur compounds dissolved therein.

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