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HYDROCARBON LUBRICANT CONTAINING HALOGEN

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No Drawing. Original application December 18, 1937, Serial No. 180,487. Divided and this application December 2, 1938, Serial No. 243,522

14 Claims. (Cl. 252—58)

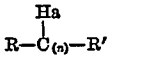
This invention is a divison of my co-pending application, Serial No. 180,487 entitled "Lubricant," filed December 18, 1937.

This invention relates to lubricants and to the method of preparing and of using the same, and is more particularly concerned with lubricants having extreme pressure characteristics.

It is well known that mineral lubricating oils can be improved by adding thereto halogenated aromatic hydrocarbons and compounds, in which hydrogen in the aromatic nucleus is substituted by chlorine. Halogenated aliphatic hydrocarbons and compounds in general have been found to be unsatisfactory because such compounds hydrolyze readily in contact with water forming acid which corrodes the metal surfaces.

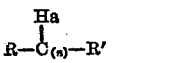
I have discovered that where halogen is linked to aliphatic carbon, which is protected on both ends by a non-acidic, stable aromatic or other carbocyclic nuclei, the resulting compounds are not only not corrosive but are more active than the halogenated aromatics. Such compounds have the ability to impart to mineral lubricating oils, increased oiliness, film strength, and extreme pressure characteristics.

The compounds which I have discovered to be suitable are those of the type



in which R and R' are non-acidic, stable aromatic or other carbocyclic nuclei, either the same or different, C represents an aliphatic radical either saturated or unsaturated having any number (n) of carbon atoms in excess of one carbon, and Ha represents a halogen united to one or more aliphatic straight chain carbon atoms which are linked between the carbocyclic nuclei.

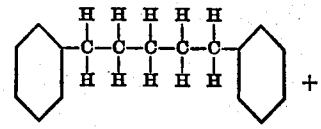
The particular compounds with which this application is concerned are those compounds of the type

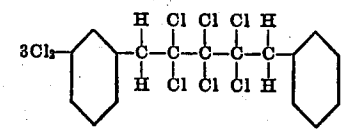


in which R and R' are benzene nuclei, C is an aliphatic hydrocarbon chain having any number (n) in excess of four carbon atoms and Ha represents a halogen united to one or more carbon atoms which are linked between the benzene nuclei.

Examples of compounds which are suitable for use according to the invention are symmetrical diphenyl pentane, symmetrical diphenyl nonane and symmetrical diphenyl nonadecane.

To illustrate graphically the nature of the compounds with which this invention is concerned: Sym. diphenyl pentane





By using 5 molecules of chlorine the hydrogen in the aliphatic chain can be entirely replaced and two atoms of chlorine attached to each carbon atom thereby obtaining decachlor symmetrical diphenyl pentane.

The halogenated compounds may be used in connection with straight mineral lubricating oils or in connection with oils which have been blended with other reagents to improve the lubricating characteristics thereof. For example, I have found that when small quantities of halogenated compounds of the nature of those above described are incorporated in a blend of mineral lubricating oil with a sulfurized and phosphorized fatty oil base such as that disclosed in the application of Whittier et al., Serial No. 55,200, the extreme pressure characteristics of such lubricants can be materially improved. A number of tests have been made on straight lubricants blended with halogenated compounds of the nature of those above set forth and on lubricants blended with sulfurized and phosphorized base together with halogenated compounds, and the results given in the table below. In these tests the straight mineral oil used was a blend in the proportions of 1:1 of two mineral lubricating oils of the following characteristics:

Table I

	Oil No. 1	Oil No. 2
A. P. I. gravity	21.5-23	17-19
Flash, ° F	345-365	510-520
Fire, ° F	385-405	590-600
Vis. at 130° F. (Say. Univ.)	98-102	1750-2100
Vis. at 210° F. (Say. Univ.)	43-45	150-165
Pour point, ° F	−20	25
Color N. P. A	2-3	8

In those tests in which an oil blended with sulfurized-phosphorized base was used, the lubricating oil, before addition of the base, had the following characteristics:

Table II

A. P. I. gravity	18–20
Flash, °F	380–400
Fire, °F	430–450
Vis. at 130° F. (Say. Univ.)	460–500
Vis. at 210° F. (Say. Univ.)	90–95
Pour point, °F	0

This oil was blended with sulfurized-phosphorized base in the proportion of 82% of oil to 18% of base.

The sulfurized-phosphorized base was made in the following manner: A homogeneous mixture of 570 pounds of Gulf Coast pale oil having a Saybolt Universal viscosity of 200 seconds and a pour point of —20° to —25° F. and 55 pounds of sulfur chloride was prepared in a cold state. This mixture was added to 9,581 pounds of prime lard oil contained in an enclosed steam jacketed kettle. The mixture was heated to 275° F. while being vigorously agitated. At this point the steam was shut off and 814 pounds of flowers of sulfur were sifted into the contents of the kettle over a period of approximately two hours. The contents of the kettle were vigorously agitated during this entire period. At the end of the two-hour period the temperature had dropped to 265° F. The steam was again turned on and the contents of the kettle heated to approximately 300° F. when the steam was turned off. Agitation was continued for a period of approximately 7½ hours after the addition of the sulfur. During this period the temperature rose from 300° to 344° F. during the first three hours indicating that an exothermic reaction was taking place. The temperature during the 7½-hour period was not permitted to drop below 320° to 330° F. and it was found that it was unnecessary to use any further heat to maintain this temperature after the mixture had been initially heated to 300° F.

The sulfurized material was then cooled to a temperature of 220° to 230° F. by circulating the material through cooling coils and back to the kettle. When this temperature had been reached, 44 pounds of phosphorous sesquisulfide was added slowly over a period of ½ hour of continuous agitation. The steam was again turned on at such times as was necessary to maintain the temperature at 220° to 230° F. After the addition of phosphorous, agitation was continued for a period of approximately five hours while the temperature was maintained at 220° to 230° F. At the end of this period the contents of the kettle were immediately cooled to 130° F. by circulation through the exteriorly located cooling coils and pumped to storage.

The following is a table of the results obtained:

Table III

Halogenated compound added	Percent added	Oil used for blend	S. A. E. test	
			1000 R. P. M.	750 R. P. M.
			Pounds	*Pounds*
		Sul-phos. base blend	90	
		Plain mineral oil lubricant	50	
Penta chlor sym. diphenyl pentane	3	Sul-phos. base blend	330	
Do	8	Plain mineral oil lubricant	120	
Do	1	Sul-phos. base blend		170
Do	2	do		550
Do	3	do		550
Do	4	do	410	550
Do	5	do	430	550
Do	1.25	do		370
Do	1.5	do		400
Do	1.75	do		435
Hexa chlor sym. diphenyl nonane	2	do		550
Octa chlor sym. diphenyl nonadecane	2	do		380

The tests were made on the SAE extreme pressure testing machine which is used in the automotive industry to determine the suitability of extreme pressure lubricants. The maximum load that can be measured on the machine with safety is between 550 and 570 pounds.

As shown in Table III, the sulfurized-phosphorized lubricant without addition of the halogen compound, withstood a load of only 90 pounds and plain mineral oil only 50 pounds. But when 4% of pentachlor symmetrical diphenyl pentane was added to a base blend of mineral oil and sulfurized-phosphorized fatty oil, the SAE test is increased beyond all expectation, a test result of 430 pounds being obtained.

The E. P. characteristics of the sulfurized-phosphorized base lubricant, as determined by the Timken machine, is not appreciably altered by addition of the halogen compound. The addition of the halogenated compound, while greatly increasing the film strength and E. P. characteristics of the lubricant, had no detrimental effect thereon. Corrosion tests made in the presence of water indicate that as much as 10% of the halogenated compound may be introduced into the lubricant without rendering it corrosive.

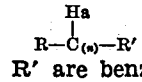
Although I do not wish to be limited to the addition of any particular amount of the halogenated compound to the lubricant, I prefer to use from 3 to 10% in connection with lubricants to be used where E. P. characteristics are important. Where the compound is added to improve the oiliness and film strength, as for example, in motor oil, the amount added may range from a fraction of 1% to approximately 5%. The upper limit of 10% has been fixed only because with the methods of testing now available, it has been found that maximum results can be obtained by adding this amount. There is no reason, however, why quantities in excess of 10% may not be used.

Instead of adding the halogenated compound directly to the oil, it may be separately fed into the particular mechanism to be lubricated and mixed in situ with the lubricating oil.

Although I have given but two specific examples of compounds which fall within the scope of my invention, it should be understood that the invention is not limited to these specific compounds, but includes compounds generally which fall within the class disclosed and claimed.

What I claim is:

1. A lubricant comprising mineral lubricating oil and a halogenated compound of the type



in which R and R′ are benzene nuclei, C is a

straight aliphatic hydrocarbon chain having any number (n) in excess of 4 carbon atoms and Ha represents a halogen united to one or more carbon atoms linked between the benzene nuclei.

2. A lubricant in accordance with claim 1 containing sulfurized and phosphorized fatty oil.

3. A lubricant in accordance with claim 1 in which the aliphatic hydrocarbon chain contains five carbon atoms.

4. A lubricant in accordance with claim 1 in which the aliphatic hydrocarbon chain contains nine carbon atoms.

5. A lubricant in accordance with claim 1 in which the aliphatic hydrocarbon chain contains 19 carbon atoms.

6. A lubricant comprising mineral lubricating oil and halogenated symmetrical diphenyl pentane in which halogen is attached to aliphatic carbon.

7. A lubricant comprising mineral lubricating oil and halogenated symmetrical diphenyl nonane in which halogen is attached to aliphatic carbon.

8. A lubricant comprising mineral lubricating oil and halogenated symmetrical diphenyl nonadecane in which halogen is attached to aliphatic carbon.

9. A lubricant in accordance with claim 6 containing sulfurized and phosphorized fatty oil.

10. A lubricant in accordance with claim 7 containing sulfurized and phosphorized fatty oil.

11. A lubricant in accordance with claim 8 containing sulfurized and phosphorized fatty oil.

12. A lubricant comprising mineral lubricating oil and pentachlor symmetrical diphenyl pentane.

13. A lubricant comprising mineral lubricating oil and hexachlor symmetrical diphenyl nonane.

14. A lubricant comprising mineral lubricating oil and octachlor symmetrical diphenyl nonadecane.

WILLIAM A. WHITTIER.