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EXTREME PRESSURE LUBRICANT

Darwin E. Badertscher and Henry G. Berger, Woodbury, and Francis M. Seger, Pitman, N. J., assignors to Socony-Vacuum Oil Company, Incorporated, New York, N. Y., a corporation of New York

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9 Claims. (Cl. 87—9)

This invention has to do in a general way with the art of lubrication and is more particularly concerned with the development of a lubricant composition adapted for use under extreme pressure conditons.

As is well known to those familiar with the art, the ordinary type of hydrocarbon lubricant, comprised only of a petroleum fraction, is incapable of withstanding the enormous pressures encountered between engaged surfaces in various types of modern machinery, such as the hypoid gears used in motor vehicles. This fact has lead to the development of the so-called "extreme pressure lubricant," which is comprised of a hydrocarbon lubricant, such as a mineral oil, containing a minor proportion of an ingredient which, under the extreme pressure conditions encountered, will provide or form between the engaged surfaces a lubricating film capable of withstanding these high pressures. Ingredients of the type just referred to are known as extreme pressure ingredients or extreme pressure (E. P.) bases, and numerous materials have been proposed for such use. Among the various E. P. bases which have been proposed and which have met with varying degrees of success are organic materials which contain sulfur and chlorine. Our invention is related to this general type of compound.

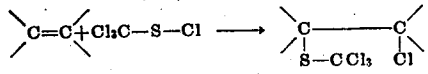
It is a primary object of this invention to provide an extreme pressure lubricant which contains a sulfur-and-chlorine-containing material or compound capable of giving to the lubricant extreme pressure properties. In this regard, we have discovered that the class or group of compounds obtained as products of reaction with perchloromethylmercaptan as the reacting reagent are very effective extreme pressure lubricant addition agents. These materials have the advantage of being cheap and easily prepared. Perchloromethylmercaptan itself and its low molecular weight derivatives are very effective E. P. bases, but we prefer to use the higher molecular weight derivatives because of the offensive odor of the former materials.

The perchloromethylmercaptan derivatives which we have found to be particularly useful as E. P. bases may be broadly divided into two general classifications, namely: (1) the products obtained by reacting perchloromethylmercaptan with materials containing reactive hydrogen atoms and reactive metal atoms, and (2) the products obtained by reacting perchloromethylmercaptan with unsaturated organic materials such as cracked petroleum stocks, etc.

Our co-pending application, Serial Number 177,891, filed December 3, 1937, is directed broadly to perchloromethylmercaptan and its derivatives as E. P. bases and specifically to those derivatives obtained by reaction with materials containing reactive hydrogen or reactive metal atoms in which the reaction is one of substitution or so-called "metathesis."

Our present invention is directed specifically to those materials obtained by the reaction of perchloromethylmercaptan with organic compounds of the unsaturated type. In other words, our invention contemplates a new extreme pressure lubricant comprised of a carrier such as hydrocarbon lubricating oil or other suitable carrying agent which contains as an extreme pressure ingredient a material obtained by the reaction of perchloromethylmercaptan with an organic material containing a double bond, such for example as unsaturated fatty materials (triolein, lard oil, cottonseed oil, linseed oil, olive oil, etc.), cracked stocks and other olefins, reformer stock, unsaturated halides such as allyl chloride, etc.

Although it is not our intention to limit the scope of our invention by any theory of reaction, we believe that the reaction of perchloromethylmercaptan with an unsaturated organic material takes place at the double bond according to the following equation:



In this way it will be seen that the substitution is made on a single molecule, as contrasted with certain other sulfochlorinating reactions, in which two or more molecules are united to form the reaction product. Our theory for the reaction exemplified by the above equation, therefore, would account for the fact that the products of reaction contemplated by this invention do not tend toward insolubility in the oil as is the case with products obtained by other sulfochlorinating reactions where the larger molecular structures are apparently formed.

Perchloromethylmercaptan, as indicated above, is a chlorinated substituted sulfur chloride having the formula, $Cl_3C—SCl$, and can be prepared by the chlorination of carbon disulfide according to the method of Helfrich and Reid (Jour. Amer. Chem. Soc. 43, 591 (1921)). For example, carbon disulfide containing about .4% by weight of iodine was chlorinated in the absence of direct sunlight at a temperature maintained below 25° C. until the volume of the reaction mixture had

doubled. This crude product, which contained carbon tetrachloride and sulfur chlorides in addition to perchloromethylmercaptan, can be used in the preparation of the E. P. bases contemplated by this invention, but it is preferable to purify the crude product by distillation on a water bath, followed by steam distillation. The material refined in this way is practically pure.

To illustrate the manner of preparing perchloromethylmercaptan derivatives of the type contemplated by this invention, namely the reaction products of perchloromethylmercaptan with unsaturated materials, the following examples are given:

(1) *Reaction with pentene-2.*—40 parts by weight of perchloromethylmercaptan and 27 parts by weight of pentene-2 were placed in a sealed container and heated to a temperature of 100° C. over a period of 23 hours. The reaction product thus formed was a dark liquid which was distilled to a liquid temperature of 145° C. under vacuum and then steam-distilled to remove unreacted perchloromethylmercaptan. The residue from the steam-distillation was separated from the water and filtered.

(2) *Reaction with lard oil.*—100 parts by weight of 70-iodine number lard oil and 58 parts by weight of steam-distilled perchloromethylmercaptan were mixed together and heated with stirring from a starting temperature of 53° C. to a final temperature of 141° C. over a period of 65 minutes. The reaction mixture was steam-distilled to remove excess perchloromethylmercaptan and the residue taken up in benzol. Alcohol was added to facilitate separation of emulsified water and the solvent was distilled off up to a liquid temperature of 115° C. under vacuum. The oil-soluble product thus obtained contained 5.60% sulfur and 16.12% chlorine.

(3) *Reaction with cottonseed oil.*—314 parts by weight of cottonseed oil and 275 parts by weight of perchloromethylmercaptan were mixed together and heated with stirring from an initial temperature of 31° C. to a final temperature of 112° C. over an interval of 1 hour. The reaction product was refined as in the preceding example.

(4) *Reaction with linseed oil.*—50 parts by weight of perchloromethylmercaptan and 25 parts by weight of linseed oil were mixed together and heated with stirring from an initial temperature of about 25° C. to a final temperature of 147° C. over an interval of 147 minutes. The linseed oil used in this experiment was rather old and probably oxidized and polymerized. The final product obtained was refined according to the procedure used in Example 2 above, and the oil-soluble refined material contained 8% sulfur and 35% chlorine.

(5) *Reaction with olive oil.*—A mixture of 252 parts of olive oil and 140 parts of vacuum-distilled perchloromethylmercaptan was heated with stirring from an initial temperature of 28° C. to a final temperature of 130° C. during 90 minutes. After refining or working up as in the preceding examples a fairly viscous oil was obtained as the reaction or condensation product, such product containing 19% chlorine and 4% sulfur.

It is to be understood that the foregoing examples are given merely by way of illustration and that the proportions of ingredients, the reaction temperatures, the intervals of reaction, and the method of refinement may be varied within relatively wide limits. It is also to be understood that the materials with which the perchlorometrylmercaptan was condensed are merely illustrative of unsaturated materials which may be used to make the perchloromethylmercaptan derivatives contemplated by this invention.

To demonstrate the effectiveness of the materials contemplated by this invention as extreme pressure lubricant ingredients we have prepared and tested various extreme pressure lubricant compositions comprised of the condensation products of perchloromethylmercaptan and the unsaturated materials described above, and hydrocarbon lubricant oil as the carrying agent. More specifically, we have prepared various lubricant blends from a mineral lubricating oil having a Saybolt Universal viscosity of 80–90 seconds at 210° F., to which a minor proportion of a product obtained by the reaction of perchloromethylmercaptan with an unsaturated material has been added. The various extreme pressure lubricants prepared in this way have been subjected to the Almen pin test (described by Wolf and Mougey, Proc. A. P. I., 1932, pages 118–130) and the S. A. E test (described in S. A. E. Journal 39, 23–4 (1936)). These two tests are commonly accepted in the art as standards to determine the extreme pressure characteristics of a lubricant.

In the table given below we show the results obtained from such tests, using the oil without the addition agent and samples of the same oil blended with the perchloromethylmercaptan derivatives of the type contemplated by this invention.

Product	Percent in oil	Extreme pressure tests (lbs. failed)	
		Almen	S. A. E. (755 R.P.M., 14.6:1 rubbing ratio, 8.35 lbs./sec. loading rate)
Oil without addition agent		3,000	40–70
Perchloromethylmercaptan condensed with:			
Lard oil	15	30,000+	580+
Do	5	11,000	325
Cottonseed oil	5	30,000+	580+
Linseed oil (emulsified, not very oil-soluble)	5	30,000+	580+
Olive oil	5	30,000+	[1] 285
Pentene-2	5	30,000	580+

[1] At 1000 R.P.M.

From the foregoing table it will be seen that the products obtained by the reaction of perchloromethylmercaptan with unsaturated organic materials are highly efficient extreme pressure lubricant bases. The amount of these materials which may be added to a hydrocarbon lubricant oil or other suitable carrying medium to form an extreme pressure lubricant composition may be varied from say .5% to 20%. It is to be understood that while we have herein described the derivatives or condensation products obtained with certain specific unsaturated materials and have illustrated exemplary methods of preparing such derivatives, the invention is not limited to the specific condensation products described nor the specific methods of preparation but includes such variations in reactants and procedures as fairly come within the scope of the appended claims. It is also to be understood that while we have referred to a hydrocarbon lubricating oil as a preferred carrying medium in the extreme pressure lubricant compositions described herein, our invention is not limited to

this particular type of carrying agent but is inclusive generally of extreme pressure lubricant compositions comprised of a condensation product of perchloromethylmercaptan and an unsaturated organic material, such product being contained in any suitable carrying agent in which it may be dissolved, dispersed or emulsified.

We claim:

1. A lubricant composition comprising a hydrocarbon lubricant oil and in admixture therewith a minor proportion of the product obtained by the reaction of perchloromethylmercaptan with an unsaturated organic material.

2. An extreme pressure lubricant comprising a hydrocarbon lubricant oil and in admixture therewith a minor proportion of the product obtained by the reaction of perchloromethylmercaptan with an unsaturated hydrocarbon.

3. An extreme pressure lubricant comprising a hydrocarbon lubricant oil and in admixture therewith a minor proportion of the product obtained by the reaction of perchloromethylmercaptan with an unsaturated fatty oil.

4. An extreme pressure lubricant comprising a hydrocarbon lubricant oil and in admixture therewith a minor proportion of the product obtained by the reaction of perchloromethylmercaptan with a material selected from the group consisting of lard oil, cottonseed oil, linseed oil, olive oil, and pentene-2.

5. An extreme pressure lubricant comprising a hydrocarbon lubricant oil and in admixture therewith a minor proportion, from about 0.5% to about 20%, of the product obtained by the reaction of perchloromethylmercaptan with an unsaturated organic material.

6. An extreme pressure lubricant comprising a hydrocarbon lubricant oil and in admixture therewith a minor proportion, from about 0.5% to about 20%, of the product obtained by the reaction of perchloromethylmercaptan with an unsaturated fatty oil.

7. An extreme pressure lubricant comprising a hydrocarbon lubricant oil and in admixture therewith a minor proportion, from about 0.5% to about 20%, of the product obtained by the reaction of perchloromethylmercaptan with a material selected from the group consisting of lard oil, cottonseed oil, linseed oil, olive oil, and pentene-2.

8. The method of lubricating metallic bearing surfaces under abnormally high pressures which comprises maintaining between said surfaces a film of hydrocarbon lubricant which contains a minor proportion of a product obtained by the reaction of perchloromethylmercaptan with an unsaturated organic material.

9. An extreme pressure lubricant comprising in combination a minor proportion of the condensation product obtained by reacting perchloromethylmercaptan with an unsaturated organic material and a major proportion of a carrying medium, such combination, when applied to metallic bearing surfaces, forming a lubricant film capable of withstanding extreme pressure conditions.

DARWIN E. BADERTSCHER.
HENRY G. BERGER.
FRANCIS M. SEGER.