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LUBRICANT AND METHOD OF PREPARING SAME

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This invention relates to lubricants and lubricant additives and to a method of preparing the same, and more particularly to the method of preparing lubricants and lubricant additives having high film strength characteristics.

One of the objects of this invention is to provide an improved method of preparing extreme pressure lubricants.

Another object is to provide an improved method of preparing a sulfurized and phosphorized extreme pressure lubricant which is non-corrosive and which does not polymerize under working conditions.

Another object is to provide a rapid method of preparing a sulfurized and phosphorized lubricant wherein the sulfurizing and phosphorizing operations are simultaneously accomplished.

Still another object of the invention is to provide an improved extreme pressure lubricant and lubricant additive containing sulfur, phosphorus and chlorine and which not only possesses exceptional film strength characteristics but which is non-corrosive and remarkably resistant to polymerization and deterioration under actual service conditions.

Efforts have been made in the past to simultaneously sulfurize and phosphorize fatty oils using phosphorus, phosphorus halides, phosphorus sulfides and various other phosphorus-containing materials as the phosphorizing agent, and sulfur, either with or without the presence of sulfur chloride catalyst, as the sulfurizing reagent. In the previous work the usual procedure was to add simultaneously, all of the ingredients to fatty oil and heat the entire mixture for varying periods of time and at varying temperatures to obtain a final lubricant. However, under these conditions it was either not possible to chemically combine a sufficient amount of the phosphorus and/or sulfur compounds to obtain the desired extreme pressure properties, or when sufficient amounts were incorporated, a heavy, polymerized mass formed which was entirely unsuitable as a lubricant or lubricant additive.

It has now been found that polymerization which occurs when elementary sulfur and phosphorus compounds are simultaneously reacted with the fatty material can be avoided and a considerable saving in time may be effected over those methods which consecutively sulfurize and phosphorize, by the carefully controlled reaction of certain sulfur compounds such as sulfur halides which react exothermically with fatty materials containing dissolved or suspended therein a suitable phosphorus compound, at a temperature below that at which polymerization of the fatty body is induced by the phosphorus compound. Free sulfur may or may not be present during the course of the reactions. However, if free sulfur is present, it is necessary to heat for longer periods of time at substantially higher temperatures than are required for the reaction of the sulfur compound, phosphorus compound and fatty body in order to complete the chemical combination of the sulfur with the fatty body and phosphorus compound.

In preparing lubricant additives in accordance with this invention, a suitable phosphorus compound such as phosphorus sesquisulfide is added to a fatty oil which may contain a minor portion of mineral oil and the mixture agitated until a uniform distribution of the phosphorus compound is obtained. The desired amount of sulfur compound such as sulfur chloride, which may preferably be diluted with mineral oil, is added to the fatty oil-phosphorus compound mixture at such rate that the heat of reaction does not increase the temperature of the mixture to a temperature above which appreciable polymerization of the fatty oil would occur, preferably not substantially in excess of 150° F. After the addition of all the sulfur chloride, agitation of the mixture is continued at a temperature not substantially in excess of 150° F. for a time usually not in excess of one hour in order that the exothermic reaction may be complete. Heat is then applied to the mixture and the temperature gradually raised over a period of approximately four to nine and one-half hours to a temperature sufficiently high, preferably not substantially in excess of 300° F., to insure chemical reaction between the fatty oil, sulfur chloride and phosphorus compound and the mixture is maintained at this temperature until the reaction has progressed to the desired stage as indicated by a copper strip corrosion test. The corrosion test consists of immersion of a polished copper strip in the reaction mixture at final reaction temperature (approximately 250° F. to 300° F.) for three minutes. A corrosion test is considered satisfactory when no black discoloration or scaling of the copper strip results. The reaction mixture is preferably rapidly cooled to a temperature below reaction temperature.

The following are specific examples of methods of preparing lubricant in accordance with this invention: 0.5% by weight of phosphorus sesquisulfide was added at room temperature to a mixture of 85% prime lard oil and 15% 100 viscosity

at 100° F. Gulf Coast neutral oil and the mixture thoroughly agitated until a uniform dispersion of phosphorus sesqui-sulfide was obtained. An amount of sulfur monochloride calculated to give 7.5% sulfur in the finished product was diluted with an equal amount of the same grade of neutral oil as was used with the lard oil, and carefully added to the lard oil-mineral oil-phosphorus sesqui-sulfide mixture, with constant agitation. An exothermic reaction occurred and the rate of addition of the sulfur monochloride was so regulated that the reaction temperature was maintained at approximately 140° F. Agitation was continued for about fifteen minutes after the final addition of the sulfur monochloride prior to increasing the temperature. At the end of this period heat was applied and the temperature gradually increased to approximately 250° to 300° F. and preferably about 250° F. to 260° F. The mixture was maintained at this temperature under conditions of constant agitation for periods of time varying from approximately four to nine and one-half hours. Samples were obtained for testing purposes at the end of four hours, six and one-half hours and nine and one-half hours total processing time. The resulting product was blended in the amount of 20% thereof by volume to 80% of S. A. E. 90 Gulf Coast mineral oil. Tests of these blends are indicated in Table I:

Table I

Sample No.	Total cooking time	Timken test		S. A. E. test	Chlorine
		Beam arm load	Pressure		
	Hours	*Pounds*	*Lbs./sq. in.*	*Pounds*	*Percent*
1	4	68	22,750	340	1.14
2	6½	77	30,300	270	0.94
3	9½	77	35,750	70	0.29
S. A. E. 90 mineral oil		18	4,500	10	

As indicated in the foregoing table, particularly by samples numbered 1 and 2, excellent extreme pressure properties were obtained as shown by both the Timken and S. A. E. tests. These tests are well known and are widely used in the industry to indicate extreme pressure properties of lubricants. A description of the procedure used in these tests may be obtained from any of a number of well known publications. It is to be noted that the first sample produced the highest S. A. E. test and lowest Timken test while the last sample, representing the longest cooking period, produced the highest Timken test and lowest S. A. E. test. It is therefore apparent that at some intermediate cooking period, as indicated by sample 2, high S. A. E. and Timken tests may be obtained. Ordinarily, a total period of cooking of approximately four to nine and one-half hours at the desired temperature is sufficient. The corrosion test on all of the foregoing samples was entirely satisfactory.

Although in the foregoing examples particular oils and compounds have been set forth for use in preparing the lubricant, it is to be understood that the invention is not limited to these specific materials. Also, any lubricating oil, paraffin, naphthene or mixed base may be used as blending stock, the only limitation being that it have specifications suitable to produce a lubricant of the quality desired. Sulfur compounds useful within the scope of this invention are those compounds which react exothermically at temperatures not substantially in excess of 150° F., with fatty material containing suitable phosphorus compounds in solution or suspension. Sulfur halides, particularly sulfur chloride, have been found to be particularly suitable. While phosphorus sesqui-sulfide has been used by way of illustration, other phosphorus compounds, including phosphorus halides such as phosphorus trichloride, oxyhalides, such as phosphorus oxychloride, sulfides such as phosphorus pentasulfide and oxides such as phosphorus pentoxide, as well as elemental phosphorus and bi-elemental phosphorus compounds such as tin phosphide may be used. The amount of sulfur compound and phosphorus yielding material used in preparing the base may vary within rather wide limits but it has been found that from 1% to 12%, and preferably 5% to 7% of sulfur by weight calculated on the finished lubricant base gives satisfactory results. The phosphorus content may vary from 0.05% to 2% and preferably from 0.1% to 0.6%. In any event and regardless of the exact amount of phosphorus and sulfur used, it is essential in order to prepare a lubricant which is satisfactory from a point of view of E. P. characteristics, corrosion and freedom from polymerization under operating conditions, to heat the materials entering into the lubricant base for a sufficient period of time to chemically combine the sulfur and phosphorus in the product. It is further essential to carefully control the temperature at which the materials are heated in order to prepare a product of the desired film strength characteristics and of uniform quality. It is particularly important that during the addition of sulfur compound the temperature not be allowed to exceed that temperature at which the phosphorus compound will either appreciably vaporize or cause an undesirable polymerization to occur.

Although lard oil was used for preparing the base in the specific example set forth, it will be understood that other fatty materials including vegetable, animal and marine oils and waxes such as cottonseed, castor, rape seed and sperm oil may be substituted in all or in part therefor. Oils of low or intermediate unsaturation are preferable to highly unsaturated oils such as linseed or tung oil, since the latter have a tendency to polymerize and do not yield products of as good E. P. characteristics as the former.

By "fatty body" as used in the specification and claims, is meant those esters commonly found in vegetable, animal and marine oils and fats, and the corresponding acids.

I claim:

1. The method of preparing a lubricant which comprises mixing with fatty body a small amount of material selected from the group consisting of phosphorus and phosphorus compound capable of reacting with said fatty body, adding sulfur halide to the mixture while maintaining the temperature below that at which appreciable polymerization occurs and subsequently heating the mixture to reaction temperature for a sufficient period of time to complete the reaction between the reagents.

2. Method in accordance with claim 1 in which the phosphorus compound is phosphorus sulfide.

3. Method in accordance with claim 1 in which the phosphorus compound is phosphorus sesqui-sulfide.

4. Method in accordance with claim 1 in which

the phosphorus compound is a halide of phosphorus.

5. Method in accordance with claim 1 in which the phosphorus compound is an oxide of phosphorus.

6. Method in accordance with claim 1 in which the sulfur halide is sulfur chloride.

7. Method in accordance with claim 1 in which the temperature is maintained not substantially in excess of 150° F. while adding the sulfur monochloride.

8. Method in accordance with claim 1 in which the reaction temperature is not substantially in excess of 300° F.

9. Method in accordance with claim 1 in which the fatty body is lard oil.

10. Method of preparing a lubricant which comprises mixing with fatty body phosphorus containing material capable of reacting with said fatty body, adding sulfur chloride to the mixture while maintaining the temperature below that at which appreciable polymerization occurs and subsequently heating the mixture to a temperature not substantially in excess of 260° F. until reaction between the reagents is completed.

11. Method in accordance with claim 10 in which the phosphorus yielding material is phosphorus sesqui-sulfide.

12. Method in accordance with claim 10 in which the phosphorus yielding material is a halide of phosphorus.

13. Method in accordance with claim 10 in which the fatty body is lard oil.

14. Method in accordance with claim 10 in which the temperature is maintained not substantially in excess of 150° F. while adding the sulfur chloride.

15. The method of preparing a lubricant which comprises preparing a mixture of phosphorus containing material capable of reacting with fatty body and lard oil, adding sulfur monochloride to said mixture while maintaining the temperature of the mixture not substantially in excess of 150° F. and subsequently heating the mixture to a temperature not substantially in excess of 260° F. until reaction between the reagents is completed.

16. A lubricant composition comprising a major portion of mineral lubricating oil and a minor portion of a material which enhances the lubricating quality of the mineral oil and prepared by mixing phosphorus containing material capable of reacting with fatty body with lard oil, adding sulfur monochloride to said mixture at such a rate that the temperature of the mixture does not substantially exceed 150° F. and subsequently heating the mixture to a temperature not substantially in excess of 300° F. until reaction between the reagents is completed.

17. A lubricant composition comprising a major portion of mineral lubricating oil and a minor portion of a material which enhances the lubricating quality of the mineral oil and prepared by mixing with fatty body, phosphorus containing material capable of reacting with said fatty body, adding sulfur monochloride to the mixture at such a rate that the temperature of the mixture does not exceed that at which appreciable polymerization occurs and subsequently heating the mixture to a temperature not substantially in excess of 300° F. until reaction between the reagents is completed.

18. The method of preparing a lubricant which comprises mixing with fatty body a small amount of material selected from the group consisting of phosphorus and phosphorus compound capable of reacting with said fatty body, adding to the mixture sulfur compound capable of reacting exothermically with the mixture at a temperature below that at which polymerization of the fatty body is induced by the phosphorus or phosphorus compound, maintaining the temperature of the mixture below that at which appreciable polymerization occurs for a sufficient period of time to complete the exothermic reaction and subsequently heating the mixture to a higher reaction temperature for a sufficient period of time to complete the reaction between the reagents.

19. Method in accordance with claim 18 in which the temperature at which the reaction mixture is maintained while the exothermic reaction is being completed is not substantially above 150° F.

20. Method in accordance with claim 18 in which the temperature at which the reaction mixture is maintained while the exothermic reaction is being completed is not substantially above 150° F. and the temperature at which the mixture is subsequently heated to complete the reaction is between approximately 250° and 300° F.

21. Method in accordance with claim 1 where the amount of phosphorus material and the amount of sulfur halide reacted with fatty body are such as to produce a finished lubricant containing about .05% to 2% of phosphorus and about 1% to 12% of sulfur.

22. Method in accordance with claim 15 where the amount of phosphorus containing material and the amount of sulfur monochloride reacted with fatty body are such as to produce a lubricant containing about .05% to 2% of phosphorus and about 1% to 12% of sulfur.

23. Composition in accordance with claim 17 where the material which enhances the lubricating quality of the mineral oil contains about .05% to 2% of phosphorus and about 1% to 12% of sulfur.

24. Method in accordance with claim 18 where the amount of phosphorus material and the amount of sulfur compound reacted with fatty body are such as to produce a lubricant containing about .05% to 2% of phosphorus and about 1% to 12% of sulfur.

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