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HEAVY-DUTY MOTOR OIL COMPOSITION

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This invention relates to lubricants and is a continuation-in-part of our application, Serial No. 424,792, filed December 29, 1941, entitled "Stabilized lubricants."

In our earlier application we disclosed lubricants and a method of preparing the same in which a fatty oil or wax, particularly woolgrease, was reacted with a phosphorus sulfide, particularly phosphorus sesquisulfide, either before or after saponification with a metal hydroxide, particularly barium hydroxide, to form a lubricant additive which when added to mineral lubricating oils imparted thereto detergent properties and lowered the pour point thereof.

We have discovered that metal soaps which have been reacted with phosphorus sulfide to form a lubricant additive can be further improved if the additive is stabilized by heating a major portion thereof to a temperature of between about 300 and 350° F., preferably about 325° F. and immediately blending it with the minor portion. This treatment serves to improve the odor of the additive and eliminate any traces of moisture that may be present.

As a preferred method of preparing our additive we saponify woolgrease or other fatty semi-wax material with a metallic oxide or hydroxide at a temperature of about 250–350° F. and then mix the saponified mixture with a phosphorus sulfide, preferably phosphorus sesquisulfide, in the presence of air at a temperature of approximately 220–230° F. for a period of about 7 to 12 hours. The mixture is then filtered to remove suspended material and then divided into two portions, one portion constituting about two-thirds of the product and the other constituting about one-third of the product by volume. The two-thirds portion is heated gradually to a temperature of about 325° F. over a period of approximately 15 to 60 minutes, depending on the volume being processed, and then immediately blended with the remaining one-third unheated portion so that the temperature of the blend will be above the boiling point of water.

As an example of a method for making our additive, one part by weight of No. 2 woolgrease was mixed with two parts of Mid-Continent mineral oil having a viscosity of about 180 seconds Saybolt and heated to 250–350° F. with sufficient barium hydroxide containing 8 molecules of water to yield a saponified product containing from 3 to 3.8% of barium (9.3 to 11.8% by weight of barium based on the woolgrease). The saponified mixture was then mixed with approximately 1.56 percent by weight of phosphorus sesquisulfide

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(5% by weight of phosphorus sesquisulfide based on the woolgrease) and the mixture subjected to agitation at a temperature of 220 to 230° F. for a period of 8½ hours with free access of air to the surface of the mixture. At the end of this period the mixture gave a good copper strip corrosion test. The mixture was allowed to cool to a temperature of about 130 to 160° F. at which temperature 2% of a commercial filter aid (Special Dicalite Speedflow) was added and the mixture filtered to remove any solid impurities contained therein. Special Dicalite Speedflow is a diatomaceous filter and prepared by calcination and contains approximately 85 per cent of silica. Two-thirds of the resulting product was heated to 325° F. over a period of about 60 minutes and immediately blended with the remaining one-third unheated portion and the blend allowed to cool. The following table gives the raw materials used in preparing the additive and the composition and properties of the finished additive.

Component	Raw Material, pounds	Finished Additive, per cent by weight
180 Vis. Mid-Continent Neutral	335.5	63.23
No. 2 Woolgrease	167.8	31.61
$Ba(OH)_2 8H_2O$	[1] 45.7	
P_4S_3	8.85	
	557.85	
Ba		3.60
S		.63
P		.93
		100.00
Specific Gravity at 60° F		0.9507
Viscosity Saybolt at 130° F		371.2

[1] 96% pure.

Sufficient of the finished additive was added to Mid-Continent S. A. E. 30 lubricating oil to give a barium content in one sample of .27% by weight and in the second sample of .20% by weight of barium and the resulting oils were subjected to a 36-hour Chevrolet Varnish test. 0.25% by weight of MS–B, a bearing corrosion inhibitor manufactured by Carbide and Carbon Chemicals Company, was also added to the oil to cut down bearing loss. MS–B is prepared by fusing together morpholine polysulfide with mercaptobenzothiazole.

In addition to the Chevrolet test, pour point determinations were made on the oils and pour stability tests were also made. Pour stability was determined in accordance with the method set forth in pages R–433 to R–436 inclusive of the

September 1, 1943, issue of the National Petroleum News in an article by L. M. Henderson and W. G. Annable. The data on the various tests is set forth in the following table.

	Per cent Ba	Per cent MS-B	Engine [1] Merit Rating	Per cent [2] Bearing Loss	Pour, ° F.	Pour Stability, [3] ° F.	
						Rapid Cycle	Slow Cycle
				(Cu-Pb Insert)			
Mid-Continent-S. A. E. 30 oil (no additive)	0	0	54	0.27; 0.25	+10	F/+20	S/−15
Mid-continent-S. A. E. 30 oil (with Additive)	0.27	0.25	92	0.41; 0.39	−15	F/−5	S/−25
Mid-continent-S. A. E. 30 oil (with Additive)	0.20	0.25	88	0.29; 0.42	−15	F/−5	S/−25

[1] 100 merit rating perfect.
[2] Bearing losses expressed in percent per whole bearing.
[3] Letters S and F under pour characteristics refer to "solid" and "fluid."

It will be evident from the data in the table that the detergency of the oil containing our additive as measured by engine merit rating is far superior to the oil without additive. As also shown in the table, the additive depressed the pour point 25° F. and the oil showed good pour stability.

It should be understood we are not limited to the use of the quantity of additive indicated in the foregoing table. We prefer, however, that the blend shall contain sufficient additive so that the finished motor oil will contain from .05 to .3% by weight of barium.

It should also be understood that we are not limited to the specific amount of barium in the saponified product. Although we prefer that the saponified product contain from about 9.3 to 11.8% of barium based on the woolgrease, satisfactory compositions have been prepared with barium contents of from 1.50 to 15.1% by weight based on the woolgrease.

It should also be understood that we are not limited to the specific amount of phosphorus sesquisulfide or to the specific amount of mineral oil used in preparing the product. The phosphorus sesquisulfide may vary from 3 to 9% based on the woolgrease and the amount of mineral oil mixed with the woolgrease may be more or less than two parts of mineral oil to one part of woolgrease, but should be sufficient to impart fluidity to the woolgrease and lower its viscosity to a point where it can be readily handled.

Other inhibitors besides MS–B may be used in conjunction with our additive in order to cut down bearing corrosion. For example, inhibitor No. 728 marketed by Lubrizol Corporation, described in Patent No. 2,261,047, is effective in conjunction with our detergent additive. Inhibitors may be used in amounts of about 0.1 to 1% by weight based on the mineral oil.

It is claimed:

1. The method of preparing a lubricant additive comprising preparing a saponified fatty semi-wax material containing in chemical combination therewith a small amount of a phosphorus sulfide, separating the resulting product into two unequal portions, heating the major portion for a short time to a temperature of about 300 to 350° F. sufficient to remove traces of moisture and objectionable odor but insufficient to decompose it and reblending it with the minor unheated portion.

2. Method in accordance with claim 1 in which the major portion is immediately combined with the unheated minor portion whereby the temperature of the blend is above the boiling point of water.

3. Method in accordance with claim 1 in which the resulting product is divided into two portions, one constituting about two-thirds and the other constituting about one-third by volume and the two-thirds portion is heated to about 325° F. and then combined with the one-third portion.

4. The method of preparing a lubricant additive comprising saponifying wool-grease with barium hydroxide so that the resulting product contains from about 1.50 to 15.1% by weight of barium based on the woolgrease, reacting the saponified woolgrease in the presence of air with an amount of phosphorus sesquisulfide equal to about 3 to 9% by weight of the woolgrease, at a temperature of about 220–230° F. for 5 to 12 hours, and stabilizing the resulting product by heating a major portion but not the entire amount thereof to a temperature of about 300–350° F. and while at said temperature combining it with the minor portion of the product.

5. Method in accordance with claim 4 in which the resulting product is separated into two portions, one constituting about two-thirds and the other one-third thereof, heating the two-third portion to about 325° F. and immediately combining it with the unheated one-third portion.

6. The method of preparing a lubricant additive which comprises saponifying woolgrease, admixed with light mineral lubricating oil, with hydrated barium hydroxide in such amount that the saponified product contains from about 3 to 3.8% by weight of barium, reacting the saponified product in the presence of air with phosphorus sesquisulfide, in an amount equal to about 5% by weight of the woolgrease, at a temperature of about 220–230° F. for 5 to 12 hours, filtering the resulting product at a temperature of about 130–160° F. and dividing the filtered product into two portions, one of which constitutes two-thirds of the total product, heating the two-thirds portion to about 325° F. for a sufficient period of time to eliminate objectionable odor and immediately combining it with the unheated one-third portion.

7. A lubricant additive comprising barium soap of woolgrease which has been chemically combined with a phosphorus sulfide and a major proportion of which has been stabilized by heat treatment at 300–350° F. for about 15 to 60 minutes.

8. A lubricant additive in accordance with claim 7 in which the woolgrease is combined with about 1.5 to 15.1% by weight of barium.

9. A lubricant additive in accordance with claim 7 in which the woolgrease is combined with about 9.3 to 11.8% by weight of barium.

10. A lubricant additive in accordance with claim 7 in which the woolgrease is combined with about 1.5 to 15.1% by weight of barium and the barium woolgrease soap is combined with from 3

to 9% by weight of phosphorus sesquisulfide based on the woolgrease.

11. A lubricant additive in accordance with claim 7 in which the woolgrease is combined with about 9.3 to 11.8% by weight of barium and the barium-woolgrease soap is combined with approximately 5% of phosphorus sesquisulfide based on the woolgrease.

12. A lubricating oil containing a small amount of a barium soap of woolgrease which has been chemically combined with phosphorus sesquisulfide, a major proportion of which soap has been stabilized by heating for approximately 15 to 60 minutes at about 300–350° F.

13. A lubricating oil containing a small amount of a barium soap of woolgrease which has been chemically combined with phosphorus sesquisulfide, a major proportion of which soap has been stabilized by heating to 300–350° F. for about 15 to 60 minutes, said soap containing from about 9.3 to 11.8% by weight of barium based on the woolgrease and from 3 to 9% of phosphorus sesquisulfide in chemical combination therewith.

14. Method in accordance with claim 1 in which the major portion constitutes approximately two-thirds of the product and it is heated for approximately 15–60 minutes at a temperature of 300–350° F.

15. Method in accordance with claim 1 in which the major portion constitutes approximately two-thirds of the product and it is heated for approximately 60 minutes at 325° F.

16. In the method of preparing a lubricant additive the steps comprising saponifying a fatty semi-wax material with barium hydroxide so that the resulting product contains from about 1.5 to 15.1% of barium based on said material and then chemically reacting the saponified product in the presence of air with phosphorus sesquisulfide, equal to 3 to 9% by weight based on said material, at a temperature of approximately 220–230° F.

17. The steps in accordance with claim 16 in which the fatty semi-wax material is wool grease.

18. The method in accordance with claim 1 in which the fatty semi-wax material is wool grease.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
2,331,923	Musselman	Oct. 19, 1943
2,130,540	Flaxman	Sept. 20, 1938
2,108,644	Brunstrum	Feb. 15, 1938
1,971,750	Kaufman	Aug. 28, 1934