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METHOD OF TREATING HYDROCARBON BASE LUBRICANTS WITH A PHOSPHORUS SULFIDE IN THE PRESENCE OF BUTYL RUBBER AND PRODUCT

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This invention relates to a process of improving hydrocarbon base lubricants and, more particularly, to an improved process of treating hydrocarbon lubricants with a small amount of a phosphorus sulfide in the presence of butyl rubber, followed thereafter, if desired, by treatment with a base, and also to the resulting improved lubricants.

Commercially used lubricants are prepared from hydrocarbon base stocks of synthetic origin or derived from natural sources such as petroleum. In order to provide a lubricant having desirable detergent characteristics lubricating oil additives customarily are added to such stocks. The solvent extracted oils, for example, are apt to be quite corrosive but this can be corrected and detergency imparted by addition of a lubricating oil additive. However, addition of such additives increases the cost of the lubricant. It would be desirable, if possible, to prepare a finished lubricant of desirable characteristics by chemical treatment of the stock.

Such a process has been described in U. S. Patent No. 2,560,543, to John D. Bartleson, dated July 17, 1951. In accordance with the disclosure of that patent, hydrocarbon base lubricating oil stock is reacted with a small amount of a sulfide of phosphorus. This reaction product can be further improved as to detergency and corrosive characteristics by reaction thereof with a base, as described, for example, in U. S. Patent No. 2,560,546 to John B. Bartleson, dated July 17, 1951. These reaction products are suitable for use under various conditions including high temperatures or high pressures or both as, for instance, in an internal combustion engine operating at high temperatures and in which the lubricant is in close contact with metallic surfaces, metal compounds and high temperature gases. They are also suitable for use in extreme pressure lubricants, e. g., in oils and greases containing the same. The treatment improves not only the corrosion, but also the viscosity increase and pentane insoluble characteristics of the oil. Only minor amounts of sludge are formed during the reaction and can be removed by filtration to produce a desirable product.

However, from time to time the sludge which forms in the course of the reaction of the phosphorus sulfide with the hydrocarbon base stock may be in the form of a tacky tar-like material which tends to adhere to the sides of the reaction vessel and which rapidly clogs the filter paper. A sticky sludge of this type also may reduce the final yield of treated oil, due to retention of the oil by the sludge. Thus, in a commercial process the poor sludge characteristics may be a serious drawback.

In accordance with the instant invention, hydrocarbon base lubricating oil stocks are reacted with a phosphorus sulfide in the presence of butyl rubber. Sludge which may form in the course of this reaction is hard and non-tacky in nature, does not adhere to the sides of the reaction vessel, does not clog the filter paper and displays little or no oil-retention characteristics. As a result, sludge readily can be separated from the desired reaction product by filtration. The butyl rubber in consequence makes possible commercial application of the process on a large scale.

The other desirable properties of the sulfide refined oil are not affected. In fact, the reaction product may have higher sulfur content and, when reacted with a base, in addition higher ash. Inasmuch as the beneficial engine

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performance characteristics of the treated oil are associated with a high sulfur and ash content, it will be appreciated that such an improvement would be notably advantageous. Further, the viscosity index of the finished oil is improved.

Some oils are quite unreactive, and do not respond readily to treatment with a phosphorus sulfide. White oil is an example. Many such oils react readily with phosphorus sulfide in the presence of butyl rubber.

The invention is of particular application to the treatment of solvent-refined hydrocarbon base stocks. These also are called solvent-extracted oils, and these terms are applied to oils treated in accordance with conventional modern methods of solvent refining lubricating oils. The oil may be a fluid hydrocarbon lubricating base stock having a viscosity at 100° F. of 10 to 500 centistokes, such as used as the base for the S. A. E. Nos. 10 to 50 oils. The oil may be obtained as a distillate or from synthetic or natural material, such as petroleum and oils produced by cracking, polymerization, dehydrogenation and the like methods. Usually the solvent selected in the treatment, such as furfural, phenol, sulfur dioxide, etc., dissolves aromatic, unsaturated and low viscosity index constituents and these are separated out. Clay treatment may or may not follow and is desirable, but not essential. Where necessary, a separate propane or the like deasphalting treatment may be used in connection with the solvent refining.

The process of the invention also is applicable to hydrocarbon base lubricant stocks obtained by other refining methods, such as acid-treated lubricant stocks, and to white oils.

The reaction of the hydrocarbon base stock with the phosphorus sulfide in the presence of butyl rubber may be conducted with direct admixture of the reactants or, if desired, by their admixture in the presence of a diluent which may subsequently be removed. Generally, a diluent is not necessary. The reaction is usually complete in about ten hours or less, and in most cases from one to two hours is an adequate reaction time. The reaction time is a function of temperature, the amount of sulfide employed, the degree of subdivision of the reactants, the efficiency of mixing and the concentration thereof in the diluent, if present.

Any phosphorus sulfide reactive with hydrocarbon base stocks can be used. Phosphorus pentasulfide (P_2S_5) is most readily available and least expensive and, therefore would most ordinarily be employed, and is used in the illustrative examples which follow. However, other phosphorus sulfides can be employed with equivalent results, such as phosphorus sesquisulfide (P_4S_3), phosphorus trisulfide (P_4S_6), phosphorus heptasulfide (P_4S_7), and phosphorus disulfide (P_2S_2).

The hydrocarbon lubricant stock is reacted with the phosphorus sulfide in the ratio of from about 0.1 to about 0.75% by weight, based on the weight of the hydrocarbon base stock, desirably about 0.25 to about 0.60%, and preferably about 0.4 to about 0.6%. Higher amounts of sulfide give products which are inferior to the hydrocarbon as to viscosity increase. Although smaller amounts than 0.1% show some improvement, at least about 0.1% is desirable.

Any amount of butyl rubber below about 2% will improve the final product. To obtain a marked improvement, at least about 0.25% butyl rubber should be present, and optimum results are obtained when amounts within the range of about 0.5% to about 1% are used. When amounts of the order of 2% and above are present, the reaction appears to be incomplete and the product is not desirable, for the sulfur and phosphorus contents are less than those of a similar product prepared using smaller amounts of butyl rubber. Evidently, excess amounts of butyl rubber inhibit the desired reaction with the phosphorus sulfide.

It is desirable to dissolve the butyl rubber in the oil before starting the reaction. This can be done by heating the oil at an elevated temperature for a sufficient time, until dissolved, and then adding the phosphorus sulfide to the resulting solution.

The reaction of the hydrocarbon base stock with the

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phosphorus sulfide in the presence of butyl rubber can be carried out in the presence or absence of air, or in an atmosphere of inert or nondeleterious gas, such as nitrogen. If an inert gas such as nitrogen is bubbled through the reaction mixture, the reaction time is reduced and the product is stabilized against liberation of hydrogen sulfide. The reaction also may be carried out under pressure, as, for example, the pressure generated through liberation of gases in the course of the reaction when the reaction is carried out in a closed vessel.

The reaction temperature varies with the hydrocarbon stock. Temperatures of at least 275° F. are desirable, while temperatures in the range of from about 300 to about 450° F. are preferred in many cases. The temperature should be below the temperature at which the reaction product or the reactants would be decomposed.

The final refined oil then can be centrifuged or filtered to remove by-products, sludge and unreacted material. If a volatile diluent is used, it can be removed by evaporation or distillation, preferably under low pressure and at low temperatures. The treated product can be used as a lubricant or in a blend together with other lubricants, lubricating oil base stocks, or lubricating agents, e. g., with soap or the like in a grease.

If desired, the reaction product can be further improved particularly with respect to detergency by reaction with a base to form a metal compound or derivative. The metal derivatives may be formed from one or more metal compounds, such as the sulfides, oxides, hydroxides, carbides and cyanamides or metals selected from groups I, II and III of the periodic table, such as potassium, zinc, barium, calcium, magnesium, sodium, strontium and aluminum. The alkali and alkaline earth metal derivatives are preferred. For particular uses, the heavier metals, i. e., those below zinc in the electromotive series, such as chromium, cadmium, tin, lead, antimony, bismuth, arsenic and the like, have been used. In the treatment with the metal compound or base, the reaction can be effected at temperatures in the range of about 100° to about 400° F., a temperature in the range of about 180° to 350° F. being preferred, if the sulfide refined stock has been subjected to a temperature of at least 300° F. Alternatively, the metal derivative may be prepared at or subjected to a temperature of 300° F.

From about 0.25 to about 15 equivalents of the metal compound may be used per mol of the sulfide used in preparing the sulfide refined stock. Preferably about 6 to about 12 equivalents are used.

The following specific examples are illustrative of the principles and advantages of the invention:

Example 1

Light white oil (600 grams) and 3 grams of butyl rubber were heated for three hours at 300° F. under a nitrogen atmosphere until the butyl rubber had completely dissolved. P₂S₅ (1.2 grams, 0.2%) was added and the reaction mixture heated for one hour at 300° F. under a nitrogen atmosphere. Substantially no sludge formed during the reaction and that which did form was hard, nontacky, and did not adhere to the sides of the flask. The product was filtered easily, a 98.5% yield being obtained, and analyzed as follows: per cent S 0.09, O. D. 238, acid No. 3.01, per cent ash 0.06, viscosity index 106.8.

The product was stable to formation of hydrogen sulfide for one week at room temperature in the absence of water.

When this oil was treated with P₂S₅ alone at 300° F. for one hour, very little reaction occurred, and most of the P₂S₅ was recovered. The product had a sulfur content of 0.025%.

Four hundred grams of the above P₂S₅-butyl rubber-oil-reaction product was reacted with 1.6 grams of flake KOH at 250° F. for two hours under a nitrogen atmosphere. Filter aid (2%) was added and the mixture filtered. The KOH appeared to have reacted completely and a 97.7% yield was obtained. This product analyzed as follows: per cent S 0.07, O. D. 47.6, alkaline No. 0.22, per cent ash 0.23, viscosity index 110.

The metal derivative was subjected to the Polyveriform test: corrosion: 7.7 mg., viscosity increase: -125 SSU at 100° F., pentane insolubles: 2.68%, neutralization No. 3.08, lacquer rating: A, sludge rating: A.

Example 2

No. 300 solvent extracted neutral oil (viscosity 300 SSU at 100° F., 600 grams) was reacted with 2.7 grams phosphorus pentasulfide at 300° F. for one hour under a nitrogen atmosphere. Very little sludge was formed, and this was hard, nontacky and readily filtered out. A yield of 97.8% was obtained after filtration.

This material (87 grams) was blended with 4.9 grams each of 78 solvent extracted bright stock (78 SSU at 210° F.) and 250 solvent extracted bright stock (250 SSU at 210° F.) to give a final lubricating oil meeting the specifications for an S. A. E. No. 20 motor oil. This material analyzed as follows: per cent S 0.39, per cent P 0.065, O. D. 90.6, acid No. 1.99, per cent ash 0.015.

To another portion (500 grams) of the above reaction product (before blending with bright stock) was added 4.5 grams of potassium hydroxide and the mixture was heated at 250° F. for two hours under a nitrogen atmosphere. The product was filtered following addition of 2% filter aid. The potassium hydroxide appeared to have reacted completely and a 97.6% yield was obtained. 27.6 grams each of 78 and 250 solvent extracted bright stock (78 and 250 SSU at 210° F. respectively) were added to give the final oil blend. The product analyzed as follows: per cent S 0.33, per cent P 0.76, O. D. 46.8, alkaline No. 0.60, per cent ash 0.35.

Example 3

No. 300 solvent extracted neutral oil (300 SSU at 100° F.), 885 grams, and 5 grams of butyl rubber were heated at 300° F. under a nitrogen atmosphere until all of the butyl rubber had dissolved. This required four hours. The solution then was mixed with 3.94 grams phosphorus pentasulfide for one hour at 300° F. in a nitrogen atmosphere. A very small amount of sludge formed which remained in suspension and was easily removed by filtration at the conclusion of the reaction. A 98.5% yield was obtained.

To 280 grams of this product were added 15.9 grams each of 78 and 250 solvent extracted bright stock to give a final lubricating oil blend which met the specifications for an S. A. E. No. 20 motor oil. The product analyzed as follows: per cent S 0.45, O. D. 231, acid No. 1.85, per cent ash 0.04, viscosity index 103.8.

Example 3A

To 600 grams of the reaction product (before blending) of Example 3 were added 5.34 grams of potassium hydroxide and the mixture reacted at 250° F. for two hours under a nitrogen atmosphere. Two per cent of filter aid was added and the product filtered. A 99.3% yield was obtained.

To this material were added 33.7 grams each of 78 and 250 solvent extracted bright stock to give a lubricating oil which met the specifications for an S. A. E. No. 20 motor oil. The product analyzed as follows: per cent S 0.37, O. D. 133.2, alkaline No. 1.05, per cent ash 0.48, viscosity index 105.2.

For comparison, oils were made without the butyl rubber as follows:

S. A. E. No. 20 blended solvent extracted neutral oil (1,000 grams) was reacted with four grams of phosphorus pentasulfide for one hour at 300° F. under a nitrogen atmosphere. Some sludge was formed in the reaction which was removed with difficulty, due to its tacky character. The product analyzed as follows: per cent S 0.41, per cent P 0.10, O. D. 108.7, acid No. 1.97, per cent ash 0.02.

To 700 grams of this reaction product was added 5.6 grams of flake KOH and the mixture reacted at 250° F. for two hours under a nitrogen atmosphere. Two per cent filter aid was added and the product was filtered. A 97.4% yield was obtained. This product had the following analysis: per cent S 0.46, per cent P 0.11, O. D. 73.5, alkaline No. 0.75, per cent ash 0.4.

Thus the products reacted in the presence of butyl rubber are fully comparable, and in alkaline number and ash slightly better than the products reacted in the absence of butyl rubber.

To show the effect of butyl rubber on viscosity index the same amount of butyl rubber was added to the initial reaction product and to the second reaction product obtained after reaction with the first product of potassium hydroxide. The viscosity index of the former was 106, and of the latter 106.7. A comparison of these results

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with the viscosity index of the products of Examples 3 and 3A shows that the improvement in viscosity index obtained when the oil is reacted with phosphorus sulfide in the presence of the same amount of butyl rubber (based on the finished oil) is approximately the same as when the butyl rubber is added to the finished oil, showing the reaction does not change the effect of butyl rubber on viscosity index.

The motor oils obtained were subjected to the Polyveriform test for 10 hours at 325° F. (Analytical Chemistry 21 737 (1949)). This is a laboratory test which is carried out under conditions found to correlate closely with the L-4 Chevrolet engine test. The following results were obtained:

Example No.	Cu-Pb Corrosion	Viscosity Increase	Neut. No.	Pentane-Insols. Mgms./10 g.	Lacquer	Sludge
Untreated S. A. E. No. 20 Motor Oil.....	19.3	170	3.92	57.7		
3.....	3.5	-30	0.95	4.8	A	A
Control No bu. rubber.....	4.3	4.5	0.62	6.4	A	A-
3A.....	4.0	-39	0.78	15.1	A	A
Control No bu. rubber.....	7.0	10.0	0.84	9.0	A	A

The Polyveriform test results show that the butyl rubber does not materially interfere with the performance of the oils. The chief difference is that the oils containing the butyl rubber give a viscosity decrease in the test, rather than a viscosity increase.

The treated oils in all cases were superior to the untreated motor oil.

Example 4

S. A. E. No. 20 motor oil (435 SSU at 100° F., 980 grams, formed by a blend of 88.5 parts of No. 300 solvent extracted neutral oil (300 SSU at 100° F.), 5 parts of 250 bright stock (250 SSU at 210° F.) and 5 parts of 78 bright stock (78 SSU at 210° F.), and 20 grams of Paratone (a polyisobutylene), and 4 grams of phosphorus pentasulfide were reacted at 300° F. for one hour under a nitrogen atmosphere. The product was filtered but a fair proportion of sticky sludge material inhibited filtration. A 96% yield was obtained of a product which analyzed as follows: per cent S 0.31, O. D. 117.2, acid No. 1.57, per cent ash 0.001, viscosity index 97.2.

To 700 grams of this product were added 5.6 grams of potassium hydroxide and the mixture reacted for two hours at 250° F. under a nitrogen atmosphere. Filter aid (2%) was added and the mixture filtered. A 98.6% yield was obtained. The product analyzed as follows: per cent S 0.30, O. D. 84.2, alkaline No. 0.65, per cent ash 0.31, viscosity index 99.6. (Example 4A in the table.)

Each of these products was subjected to the Polyveriform test with the following results:

Example No.	Cu-Pb Corrosion	Viscosity Increase	Neut. No.	Pentane-Insols. Mgms./10 g.	Lacquer	Sludge
Untreated S. A. E. No. 20 Motor Oil.....	19.3	170	3.92	57.7		
4A.....	16.1	9.0	1.08	13.4	A	A
.....	7.0	14.0	1.55	12.6	A	A

These results show that the presence of Paratone did not improve the product in any way. The sludge which formed with the Paratone present was of the same type as that which formed in the absence of butyl rubber. Also, a much lower viscosity index improvement is obtained and the engine performance characteristics are not good, as a comparison of Examples 3 and 4 and 3A and 4A, respectively, shows.

Example 5

No. 300 solvent extracted neutral oil (300 SSU at 100° F., 885 grams) and 5 grams of butyl rubber were

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heated at 300° F. under a nitrogen atmosphere until all of the butyl rubber had dissolved. The solution was heated with 1.97 grams phosphorus pentasulfide and 300° F. under a nitrogen atmosphere for one hour. A very small amount of hard, nontacky sludge was formed which readily was removed by filtration. A 98.5% yield was obtained.

To 280 grams of the product was added 15.9 grams each of 78 and 250 solvent extracted bright stock to give a motor oil meeting the specifications for an S. A. E. No. 20 motor oil. This product analyzed per cent S 0.26, O. D. 174.8, acid No. 1.3, per cent ash 0.007, viscosity index 105.

To 600 grams of the above product (before blending)

was added 2.67 grams of potassium hydroxide, and the mixture reacted for two hours at 250° F. under an inert atmosphere. Filter aid (2%) was added and the product filtered, a yield of 97% being obtained.

Motor oil which met S. A. E. No. 20 motor oil specifications was prepared by blending the above product with 33 grams each of 78 and 250 solvent extracted bright stock. The product analyzed per cent S 0.26, O. D. 84.7, alkaline No. 0.61, per cent ash 0.22, viscosity index 108.7.

Example 6

Light white oil (600 grams) and 6 grams of butyl rubber were heated for three hours at 300° F. under a nitrogen atmosphere until the rubber was completely dissolved. To this solution was added 2.4 grams P_2S_5 and the mixture reacted at 300° F. for one hour with nitrogen stripping. A small amount of hard, non-tacky sludge formed which was readily separated by filtration. The product analyzed per cent S 0.18, per cent P 0.08, O. D. 258, acid No. 2.0, per cent ash 0.02.

Example 6A

To 500 grams of this reaction product was added 4 grams of flake potassium hydroxide and the mixture heated at 250° F. for two hours with nitrogen stripping. The mixture was filtered with the aid of 2% filter aid. The potassium hydroxide appeared to have reacted completely and the reaction product analyzed per cent S 0.09, per cent P 0.05, O. D. 108, alkaline No. 0.97, per cent ash 0.43, viscosity index 113.55.

These reaction products were subjected to the Polyveriform test, the results of which are reported below:

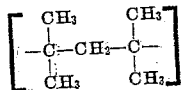
Example No.	Corrosion, mg.	Vis. Increase, SSU at 100°	Pentane ins., percent	Neut. No.	Lacquer	Sludge
6.....	136.8	18.4	0.45	1.06	A	A-
6A.....	43.2	250.4	1.78	5.77	A	A

7 Example 7

Light white oil (1400 grams) and 28 grams of butyl rubber were heated for three hours at 300° F. with nitrogen stripping until the rubber was completely dissolved. To 1200 grams of this solution was added 4.8 grams (0.4%) P₂S₅ and the mixture reacted at 300° F. for one hour with nitrogen stripping. A small amount of crystalline sludge formed which was readily separated by filtration. The product analyzed percent S 0.03, percent P 0.03, O. D. 7.3, acid No. 1.14, percent ash 0.02, viscosity index 131.4. This shows that no appreciable reaction occurred.

To 600 grams of this reaction product was added 4.8 grams of flake potassium hydroxide and the mixture heated at 250° F. for two hours with nitrogen stripping. The mixture was filtered with the aid of 2% filter aid. The potassium hydroxide appeared not to have reacted, and was recovered upon filtration. The reaction product analyzed percent S zero, percent P 0.03, O. D. 15.6, acid No. 0.44, percent ash 0.09, viscosity index 133.3, showing that substantially no reaction occurred.

Butyl rubber, as the term is used in the claims, refers to GR-1, the product of the copolymerization of isobutene with a small amount of isoprene or butadiene, and this is known to have the chain structure:



and contains butadiene or isoprene unsaturated units at random intervals in the chain. Butyl rubber normally is estimated to contain about 5% of these unsaturated units. It is thought that the butyl rubber takes part in the phosphorus sulfide hydrocarbon oil reaction by reaction with the phosphorus sulfide or the sulfide-oil reaction product at the unsaturated carbon-to-carbon double bonds. Reaction of some type undoubtedly occurs, as is evidenced by the different type of sludge, hard and nontacky rather than tarry in character, which forms in the presence of the butyl rubber.

The theory is further verified by the inactivity of Paratone. Paratone is a butene polymer which is fully saturated, and it would be expected if the above theory were correct that it would be unable to take part in the reaction because of the absence of carbon-to-carbon double bonds. This in fact has been shown to be the case. Buna S, a styrene-butadiene polymer, and Buna N, an acrylonitrile polymer, are insoluble in oil and, likewise, appear to be ineffective in the processes of the invention. Completely vulcanized butyl rubber, likewise, is oil insoluble and is not employed. Unvulcanized or partially vulcanized butyl rubbers are employed in the process of the invention.

If desired, the improved lubricants of the invention may be used in blends together with other lubricants or lubricant agents, e. g., with soap or the like in a grease. If desired, an agent for improving the clarity of the oil may be included, e. g., lecithin, lauryl alcohol and the like. If desired, an agent for preventing foaming may be included, e. g., tetra-amyl silicate, an alkyl ortho-carbonate, ortho-formate or ortho-acetate or a polyalkyl silicone oil.

All parts and percentages in the specification and claims are by weight.

We claim:

1. A method of processing hydrocarbon lubricating oil stock which method comprises treating said stock with an amount of phosphorus pentasulfide in the range of about 0.1 to about 0.75% at a temperature in the range of about 275° to about 450° F. in the presence of a small amount within the range from about 0.25 to 2% of butyl rubber.

2. A composition suitable for use as a lubricant and as a lubricant additive obtained by the process of claim 1.

3. A method of processing hydrocarbon lubricating oil stock to yield an oil having improved service properties which method comprises treating said stock with an amount of phosphorus pentasulfide in the range of about 0.1 to about 0.75% at a temperature in the range of about 275° to about 450° F. in the presence of a small amount within the range from about 0.25 to 2% of butyl rubber and then with an amount of a base in the range of about 0.25 to about 15 equivalents per mol of the phosphorus sulfide.

4. A composition suitable for use as a lubricant and as a lubricant additive obtained by the process of claim 3.

5. The method of claim 1 wherein the stock is treated with an amount of phosphorus pentasulfide in the range of about 0.25 to about 0.6% at a temperature in the range of about 300° to 450° F.

6. A composition suitable for use as a lubricant and as a lubricant additive obtained by the process of claim 5.

7. The method of claim 3 wherein the stock is treated with an amount of phosphorus pentasulfide in the range of about 0.25 to about 0.6% at a temperature in the range of about 300° to 450° F. and then with an amount of a metal base in the range of about 1.0 to about 15 equivalents per mol of the phosphorus pentasulfide.

8. A composition suitable for use as a lubricant and as a lubricant additive obtained by the process of claim 7.

9. The method of claim 7 wherein the stock is treated with an amount of dry potassium hydroxide in the range of about 0.6 to about 0.8% by weight of the stock.

10. A composition suitable for use as a lubricant and as a lubricant additive obtained by the process of claim 9.

11. A method of processing hydrocarbon lubricating oil stock to yield an oil having improved service properties which method comprises treating said stock with an amount of phosphorus pentasulfide in the range of about 0.1 to about 0.75% at a temperature in the range of about 275° to about 450° F. in the presence of a small amount within the range from about 0.25 to 2% of butyl rubber and then with a base in the amount of at least about 0.25 equivalent per mol of the phosphorus pentasulfide.

12. A composition suitable for use as a lubricant and as a lubricant additive obtained by the process of claim 11.

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