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METHOD OF IMPROVING LUBRICANTS AND LUBRICATING OIL ADDITIVES

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This invention relates to lubricants and lubricant additives suitable for use under various conditions, including high temperatures or high pressures, or both, as for example, use in an internal combustion engine operating at higher temperatures and in which the lubricant is in close contact with metallic surfaces, metal compounds and high temperature gases. Lubricating oils, particularly when used under such conditions, are subject to breakdown resulting in lacquer and varnish deposition, sludge and acid formation, and corrosion of the bearing and other materials with which they are in contact.

This application is a continuation-in-part of our application Serial No. 578,122, filed February 15, 1945, now abandoned.

Various phosphorus sulfide derived lubricating oil additives have been proposed heretofore, and among these are the reaction products of a phosphorus sulfide with an olefinic hydrocarbon.

These additives are well known in the art. In general, they are made by reacting any olefin of widely varying molecular weight, with 1 to 50% phosphorus sulfide, such as P_2S_5, at a temperature of 200° to 500° F. and separating the reaction product from any sludge that may be formed. The art understands these phosphorus sulfide-olefin type additives and further detailed description is unnecessary in view of this understanding in the art and the illustrative examples set forth hereafter.

It has now been found that these phosphorus sulfide lubricant additives can be markedly improved as to their solubility, color, odor, lubricant characteristics, or combinations thereof.

As illustrative of the invention, a phosphorus sulfide-olefin substrate, such as the reaction product of phosphorus pentasulfide with an olefin, is mixed with a non-polar solvent, such as pentane, which is partially miscible and only partially miscible with the substrate to form two phases, an extract phase and a raffinate phase. The phases are separated and the extract recovered from the extract phase. The solvent may be recovered and may be reused in the process. By this means, the extract of the phosphorus sulfide derived lubricant additive is obtained; and it has a preponderance of the characteristics desired for a lubricant or a lubricant additive, i. e., is superior to the substrate. Since it is assumed that the portion insoluble in a solvent, such as pentane, is also insoluble in oil, the extract phase has better oil solubility. It also has superior color and an oil containing the extracted additive is free from sediment, has good color, and has improved resistance to deterioration.

The di-phasic solvent extraction process of the invention involves contacting the substrate with a non-polar substance in which at least one of the components of the substrate is soluble to

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some extent and in which the solubilities of the various components of the substrate are different, so that the system of solvent and substrate forms into two phases, the extract phase being richer in the more soluble components, and the raffinate phase being richer in the less soluble components of the original substrate; and, as far as we are aware, it has never been proposed heretofore to so process these phosphorus sulfide derived lubricant additives and it has never been shown that any advantage would result therefrom.

The new extraction process may be conveniently carried out in a continuous countercurrent fashion or in a batch operation or a sequence of batch type extractions. Generally, the solvent will be a liquid and employed at temperatures and pressures at which it has a maximum solvent effect; e. g., at or near its boiling point, or at other temperatures to advantage, including at elevated temperatures and pressures. The solvent employed is selected in accordance with the desired solvent power. Mixtures of solvents may be used. The non-polar solvents include ether, hydrocarbons (including lower hydrocarbons having not over eight carbon atoms in the molecule), such as, liquid propane, pentane, naphtha, and non-polar chlorinated hydrocarbons, such as, carbon tetrachloride, and the like. The non-polar solvents, particularly the hydrocarbons are relatively inexpensive and readily available. Also, since compounds insoluble therein are assumed by the art to be oil insoluble, the invention may be used to prepare an additive that is completely oil soluble.

The improved lubricants and lubricant additives obtained in accordance with the invention may be used similarly to the substrate lubricant additive as disclosed hereinbefore. Generally, however, as an additive, a lower concentration of the extract may be used to advantage.

The following examples of the preparation of the improved materials in accordance with the invention, and tables of results of tests of lubricants comprising some of such improved compositions, will serve to illustrate and point out some advantages, but none of the examples or embodiments are to be in any wise construed as limiting the scope of the invention as otherwise disclosed and claimed herein.

EXAMPLE 1

(a) A commercially desirable additive may be prepared by reacting so-called "motor polymer" or "reduced motor polymer" with a sulfide of phosphorus. Motor polymer is usually made from C_3 and C_4 olefins by non-selective polymerization, e. g., with a phosphorus acid type catalyst. Motor polymer is composed of mono-olefinic hydrocarbons and boils in the range of 80° to 500° F.,

with a major portion boiling in the range of 120° to 400° F. A polymer gasoline fraction may be removed therefrom by fractional distillation to the 250° F. cut point, and this reduced form motor polymer is particularly useful. The reduced motor polymer may contain small amounts of materials lighter than the trimer. Its average molecular weight is about 145 and it preferably contains a major amount of branched chain olefins boiling below 600° F.

38 parts of P_2S_5 is mixed with 62 parts of reduced motor polymer and agitated for two hours at 350° F. in a pressure reaction vessel, at a pressure of about 100 pounds per square inch. Some gas is vented during the reaction, depending upon the free space in the vessel, in order to maintain this pressure. A 100° yield is obtained, based on the motor polymer, and no sludge is formed, but it is preferred to filter the reaction product. The product has an optical density of 31.8 and contains 30.7% by weight of pentane insolubles. This product is referred to as additive 1 (*a*) hereinafter.

Portions of this reaction product 1 (*a*) are shaken with n-pentane in the ratios indicated hereinafter, allowed to stand two hours, and then centrifuged. The upper layer (extract phase) is separated from the lower layer (raffinate phase), and then the solvent is distilled from the extract in the extract phase. The amount of extract obtained is as follows (and the particular extract is referred to hereinafter by the example number):

Example Number	c. c. of Example 1 (*a*) Product (i. e., Substrate)	c. c. of n-Pentane	Extract in Weight Per Cent of Substrate
1 (*b*)	20	80	68.7
1 (*c*)	30	70	67.6
1 (*d*)	50	50	84.9
1 (*e*)	70	30	98.5

The above examples were tested by the "Sohio" Corrosion Test. This test is described in a copending application of E. C. Hughes, J. D. Bartleson, M. L. Sunday and M. M. Fink, which also correlates the results of the laboratory tests with a Chevrolet Engine Test.

Essentially the laboratory test equipment consists of a vertical thermostatically heated glass test tube (45 mm. outside diameter and 42 cm. long), into which is placed the corrosion test unit. An air inlet is provided for admitting air into the lower end of the corrosion unit in such a way that in rising the air will cause the oil and suspended material therein to circulate into the corrosion unit. The tube is filled with an amount of the oil to be tested which is at least sufficient to submerge the metals being tested.

The corrosion test unit essentially consists in a circular relatively fine grained copper-lead test piece of $\frac{1}{16}$" O. D., which has a $\frac{1}{4}$" diameter hole in its center (i. e., shaped like an ordinary washer). The test piece has an exposed copper-lead surface of 3.00 sq. cm. Of this surface area, 1.85 sq. cm. acts as a loaded bearing, and is contacted by a part of the cylindrical surface of a hardened steel drill rod (14" diameter and $\frac{3}{32}$" long, and of 51–57 Rockwell hardness).

The drill rod is held in a special holder, and the holder is rotated so that the surface of the drill rod which contacts the bearing sweeps the bearing surface (the drill rod is not rotated on its own axis and the surface of the drill rod which contacts the bearing is not changed).

The corrosion test unit means for holding the bearing and the drill rod is a steel tubing (15" long and $1\frac{1}{32}$" O. D.) which is attached to a support. A steel cup (1" long, $1\frac{1}{32}$" O. D. by $\frac{13}{16}$" I. D.) is threaded into the steel tube, at the lower end. The cup has a $\frac{3}{8}$" diameter hole in the bottom for admitting the oil into the corrosion chamber. The copper-lead test piece fits snugly into the steel cup and the hole in the test piece fits over the hole in the steel cup. A section of steel rod ($\frac{3}{8}$" in diameter and 19" long) serves as a shaft and is positioned by 2 bearings which are fixedly set in the outer steel tubing, one near the top and one near the lower (threaded) end thereof. Several holes are drilled just above and just below the lower bearing. The holes above the bearing facilitate cleaning the apparatus, while the holes below the bearing enable the circulation of oil through the corrosion chamber. The drill rod holder is connected to the shaft by a self-aligning yoke and pin coupling. This assures instantaneous and continuous alignment of the drill rod bearing member against the bearing surface at all times. A pulley is fitted to the top of the steel shaft and the shaft is connected therethrough to a power source. The shaft is rotated at about 675 R. P. M.; and the weight of the shaft and attached members is about 600 grams, which is the gravitational force which represents the thrust on the bearing. The air lift from the air inlet pumps the oil through the chamber containing the test piece and out through the holes in the steel tubing.

The ratios of surface active metals to the volume of oil in an internal combustion test engine are nearly quantitatively duplicated in the test equipment. The temperature used is approximately that of the bearing surface. The rate of air flow per volume of oil is adjusted to the same as the average for a test engine in operation. Of the catalytic effects, those due to soluble iron are the most important. They are empirically duplicated by the addition of a soluble iron salt. Those due to lead-bromide are duplicated by its addition.

The test was correlated with the L–4 Chevrolet test, and a slightly modified version of this test. The modified test comprised reducing the oil additions from the 4 quarts in the usual procedure to 2 quarts, by reducing the usual 1 pint oil additions which are made at 4 hour intervals to ½ pint additions. This modification increases the severity of the test in its corrosion and detergency components, particularly in the case of border line oils.

For each test, the glass parts are cleaned by the usual chromic acid method, rinsed and dried. The metal parts are washed with chloroform and carbon disulfide and polished with No. 925 emery cloth or steel wool. A new copper-lead test piece is used for every test. The test piece is polished before use, on a surface grinder to give it a smooth finish. The test piece is weighed before and after the test on an analytical balance to evaluate the corrosion. After placing the oil and corrosion test unit in the tube, and bringing the assembly up to temperature in the thermostat, soluble catalyst is added and the air flow is started. Lead bromide catalyst is added immediately after starting the air, and timing of the test is begun.

The laboratory test conditions which were

found to correlate with the Chevrolet procedure 36-hour test are shown in the following table:

Table A

Temperature	325° F.
Oil sample	107 cc.
Air flow rate	70 liters/hour.
Time	10 hours.
Catalyst	Steel. Copper - lead bearing: 3 sq. cm. area of which 1.85 sq. cm. is a bearing surface.
	Ferric 2 - ethyl hexoate—0.05% as Fe_2O_3 in C. P. benzene
	Lead bromide—0.1% as precipitated powder
Bearing assembly:	
Load	600 grams.
Speed	675 R. P. M.

By extending the laboratory test to 20 hours, it was found that correlation with the Chevrolet 72-hour test could be obtained.

At the close of the test period, the extent of corrosion is determined by reweighing the corrosion test piece and determining the change in weight due to the test. An accurate evaluation of the lacquering properties of an oil is obtained by a visual rating system which is applied to the outer surface of the corrosion unit steel tube and metal cup in much the same way that the piston skirt, cylinder wall, etc., of an engine are rated for varnishes. The sludge rating of the engine is simulated by a visual rating of the insoluble materials and used oil which are coated on the glass test tube at the conclusion of the test. For both sludge and varnish rating a scale rating of A (best) to F (worst) is used.

A sufficient volume of used oil is obtained from the test for determination of the usual used oil properties.

The "Sohio" Corrosion Test was run on blends of conventionally acid treated Mid-Continent oil (S. A. E. 20) and the above additives at 325° F. for 20 hours; the results given in Table (A) are representative.

Table (A)

Additive from Example No	1 (*a*)	1 (*b*)	1 (*c*)	1 (*d*)	1 (*e*)
Concentration of Additive (in per cent by weight)	1.5	1.5	1.5	1.5	1.5
Sludge (isopentane insoluble—in milligrams)	5.65	3.56	4.46	3.83	4.35
Corrosion (milligrams per sq. cm. weight loss of Cu-Pb)	5.5	57.4	7.5	3.3	35.1
Viscosity Increase (SUS)	232	120	122	117	117
Sludge Rating	B	A	B	B	B
Lacquer Rating	C	B	B	B	B

The same test was also run on blends of a conventionally solvent extracted lubricating oil and the above additives; the results given in Table (B) are representative.

Table (B)

Additive from Example No	1 (*a*)	1 (*b*)
Concentration of Additive (in per cent by weight)	1.5	1.5
Sludge (isopentane insoluble—in milligrams)	1.69	0.72
Corrosion (milligrams per sq. cm. weight loss of Cu-Pb)	113.2	6.4
Viscosity Increase (SUS)	76	62
Sludge Rating	B+	A
Lacquer Rating	B	B

The above data show that improved additives are obtained by the process of the invention; i. e., that the extract is superior to the substrate as an additive. With the acid treated oil (Table (A)), the 1 (*d*) additive shows particularly low corrosion; the 1 (*b*) additive shows low viscosity increase and improved sludge and lacquer rating. The ratio of solvent can be selected in accordance with the properties wanted in the extracted additive and the yields. With the solvent extracted oil (Table (B)), the 1 (*b*) additive shows improvement in substantially all characteristics; and a particularly noteworthy improvement in the corrosion (with this type of oil, corrosion is the most important problem).

EXAMPLE 2

(*a*) The same reaction product as 1 (*a*) is used herein.

(*b*) 30.5 weight per cent of this reaction product, 2 (*a*), are mixed with 65.9 weight per cent of liquid propane in a stainless steel pressure vessel, shaken, and then allowed to settle, whereby two phases form. The upper propane layer (extract phase) is separated from the lower layer (raffinate phase). The solvent is removed from the extract phase, and is referred to as 2 (*b*).

The "Sohio" Corrosion Test was run on blends of conventionally solvent extracted lubricating oil and the above additives for 20 hours at 325° F.; the results given in Table (C) are representative.

Table (C)

Additive from Example No	2 (*a*)	2 (*b*)
Concentration of Additive (in per cent by weight)	1.5	1.5
Sludge (isopentane insoluble—in milligrams)	1.68	0.92
Corrosion (milligrams per sq. cm. weight loss of Cu-Pb)	113.2	35.4
Viscosity Increase (SUS)	76	167
Sludge Rating	B+	A
Lacquer Rating	B	B+

This data shows that the extract 2 (*b*) obtained in accordance with the invention gives a superior blend with a solvent extracted oil, as compared to the substrate 2 (*a*); and the marked reduction in corrosion is especially noteworthy.

EXAMPLE 3

(*a*) The same reaction product as 1 (*a*) is used herein.

Portions of this reaction product 3 (*a*) are shaken with virgin (straight run) naphtha, allowed to settle, whereby two layers form. The upper naphtha layer (extract phase) is separated from the lower layer (raffinate phase). The solvent is distilled from the extract phase. The relative amounts of substrate and solvent, and the amount of extract obtained is indicated in the following (the extract is referred to hereinafter by the example number):

Example Number	c. c. of Example 3 (*a*) Product (i. e., Substrate)	c. c. of Naphtha	Extract in Weight Per Cent of Substrate
3 (*b*)	10	90	88.7
3 (*c*)	20	80	88.9

The "Sohio" Corrosion Test was run on blends of conventional solvent refined oil and the above additives for 20 hours at 325° F.; the results given in Table (D) are representative.

Table (D)

Additive from Example No.	3 (a)	3 (b)	3 (c)
Concentration of Additive (in per cent by weight)	1.5	1.5	1.5
Sludge (isopentane insoluble—in milligrams)	1.68	0.75	0.77
Corrosion (milligrams per sq. cm. weight loss od Cu-Pb)	113.2	57.4	48.2
Viscosity Increase (SUS)	76	66	81
Sludge Rating	B+	A−	A
Lacquer Rating	B	B	B

This data also shows that an improved additive (extract) is obtained in accordance with the invention. The improvement in corrosion of the 3 (*b*) and 3 (*c*) blend, as compared to the 3 (*a*) blend, is particularly noteworthy, especially in connection with a solvent extracted oil where corrosion is a problem.

The examples described heretofore are concerned with the extraction of the sulfide-olefin additive by means of a non-polar solvent. This is selected for the purposes of illustrating the best mode of carrying out the invention because of the low cost of the additive and the non-polar solvent.

The invention, however, is not limited to the use of a non-polar solvent and a polar solvent may also be used. For example, the same sulfide-olefin reaction product described in Example 1 (*a*) may be extracted with nitromethane, which is an illustrative example of a polar solvent.

EXAMPLE 4

20 parts by weight of the sulfide-olefin reaction product of Example 1 (*a*) is agitated with 80 parts of nitromethane and it is found that 76.1% of the sulfide-olefin reaction product is soluble in the nitromethane. The nitromethane is removed from the extract phase and the extract remaining is a superior lubricating oil additive as compared with the substrate.

In the above examples, the invention has been described primarily in connection with the extraction of a sulfide-olefin reaction product as the substrate because of the low cost of the olefin as a raw material. The invention, however, is not limited thereto and is applicable to any of the sulfide-derived reaction products. Another class of such materials which have found wide use as lubricating oil additives consists of the reaction product of a phosphorus sulfide and an ester type wax, such as, degras, sperm oil, lanolin and similar oxygen-containing waxes.

The following example is given as illustrative of such an embodiment of the invention:

EXAMPLE 5

(*a*) 600 grams of neutral grade degras and 150 grams of P_2S_5 were mixed and heated to 330° F. for three hours with agitation and then maintained at this temperature for an additional hour without agitation. 5.4 grams of gas were evolved. The reaction mass was filtered and the filtrate blown with nitrogen while maintained at 212° F. for one hour. 557.0 grams of a black P_2S_5-degras reaction product and 187.5 grams of sludge were obtained. The reaction product analyzed 3.48% by weight ash, and 13.31% S.

(*b*) 37.15 grams of the above product was extracted with 4,000 cc. of boiling acetone in steps using 20 to 200 cc. at a time. The raffinate or acetone insoluble material was a very viscous black resin which analyzed 36.05% S. It weighed 4.25 grams, that is, 12.5% by weight of the charge.

(*c*) The acetone extract phase was filtered and the acetone distilled off. 32.8 grams of extract of acetone soluble material was obtained. This is 37.5% by weight of the charge. It was a non-viscous light red colored oil which analyzed 10.5% S.

(*d*) 84 grams of P_2S_5-degras product (*a*) and 16 grams of barium hydroxide octahydrate were mixed and heated at 190° F. with agitation for four hours and then to 250° F. for one hour. The reaction product was centrifuged hot. 76 grams of very viscous orange oil was obtained as the product (barium-P_2S_5-degras). It analyzed 8.57% by weight ash, and 11.52% S.

(*e*) 25.45 grams of the above extract from (*c*) and 6.36 grams of barium hydroxide octahydrate were mixed and heated with agitation for four hours at 190° F. and then to 250° F. for one hour. The reaction product was dissolved in ether and filtered hot. The ether was distilled off from the filtrate 25.25 grams of a very viscous light red colored oil which analyzed 24.36% by weight Ba and 11.33% S was obtained.

In order to compare the efficiencies of these different additives, lubricating oil compositions containing the improved additives were made up and tested according to laboratory test procedures for evaluating the service stability of oils, as described in a paper by R. E. Burk, E. C. Hughes, W. E. Scovill and J. D. Bartleson presented at the Atlantic City Meeting of the American Chemical Society in September 1941, and in another paper by the same authors presented at the New York City Meeting of the American Chemical Society in September 1944. The latter paper also correlates the results of such laboratory tests with the so-called "Chevrolet Engine Test." This test is described in Patent No. 2,403,894 at column 8, line 42, et seq. including the conditions for the "Standard Test."

"Standard" tests were run on compositions containing a conventional Mid-Continent acid treated lubricating oil base stock blended with Pennsylvania bright stock (S. A. E. 30) and the above additives of Example 5.

The following data is given:

	Concentration	Engine Appearance Rating	Color
	Per cent	*Per cent*	
Blank Oil	0	54.3	Light Amber.
With Additive 5 (*a*)	1.5	82.5	Black.
With Additive 5 (*b*)	0.19	62.3	Do.
With Additive 5 (*c*)	1.31	90.5	Clear Light Red.

In the tests, the extract (*c*) gives a better engine appearance rating than the substrate (*a*) when used in a smaller amount, as an additive, i. e., the corresponding amount which is extracted. A comparison of 5 (*b*) and 5 (*c*) shows that the good part of the 5 (*a*) additive is in the 5 (*c*) fraction. The extract is a clear red color as compared with the opaque blackness of the substrate; this gives an oil of light color and is particularly advantageous from the customer acceptance viewpoint.

The following data is also given:

	Concentration	Engine Appearence Rating	Color
	Per cent	*Per cent*	
Blank Oil	0	54.6	Light Amber.
With Additive 5 (*a*)	1.5	82.5	Black.
With Additive 5 (*d*)	1.5	88.0	Do.
With Additive 5 (*e*)	0.5	86.8	Clear Light Red.

Preparation of a metal derivatives from the extract (*c*) is illustrated by (*e*), and when this is used as an additive, about the same improvements in used oil rating and engine appearance rating are obtained from only one third of the amount of (*e*) as from the corresponding metal derivatives of (*d*). In this method of preparing the metal derivative, a saving of chemicals is also achieved, since the metal compound is not used to treat the undesirable fraction of the substrate. Also, the oil to which the additive 5 (*e*) has been added is of better color.

The invention is not limited to the improvement by selective solvent extraction of substrates made by reaction with the above-described materials, nor to any particularly selective solvent. The art well understands that a phosphorus sulfide can be reacted with a wide variety of materials to form an additive by imparting certain desirable lubricating properties to oil. Examples of these are the reaction products of a phosphorus sulfide with nitriles, amides, terpenes, aromatic compounds, naphthalene type compounds, amines, oxygen-containing compounds, such as fatty acids, esters of fatty acids, ketones, alcohols, etc. Since the invention is not concerned with the manufacture of these reaction products, per se, and they are known in the art, it is believed unnecessary to describe them in greater detail. The following table illustrates the various types of materials which can be reacted with a phosphorus sulfide to obtain a substrate which can be improved by extraction. In the left hand column is illustrated the additive made by reacting the listed materials with phosphorus sulfide and in the right hand column is listed the selective solvent which may be used in extracting the substrate. In all instances, the relative amounts, temperatures, etc., will be selected so as to give the amount of the extract having the desired properties.

Reactant with Phosphorus Sulfide	Selective Solvents
Cetyl Palmitate	Ether
Hydrogenated Sperm Oil	Methylethyl Ketone
Octadecane Nitrile	Ethane
Dodecane Nitrile	Isophorone
Alpha Pinene	N-butane
Terpineol	Mesityl Oxide
Cyclohexanol	Furfural
Capryl Alcohol	Dioxane
Tetradecanol	Diethyl Sulfide
Octadecaneamide	Thioacetone
Tetradecaneamide	Isopentane
Xylylheptadecyl Ketone	Sulfur Dioxide
Dipalmityl Ketone	Methyl Isobutyl Ketone
Lauric acid	Propionaldehyde
Oleic acid	Nitroethane
Coconut Fatty Acids	Ethylacetate
Dioctadecylamine	Methylcellosolve
Octadecene-1	Isopropanol
Lauryl Mercaptan	Propyl Mercaptan

When it is desired to make a corresponding metal compound, the extract which has been separated by the selective solvent action may be reacted with a desired metal, such as, an alkali metal, alkaline earth metal or heavier metals, such as, aluminum, arsenic, etc., to give a metal derivative, which is superior to the metal derivative of the substrate paragraph.

Following comparable procedures, any oil-soluble reaction product of an organic compound with a sulfide of phosphorus, which reaction product is a suitable additive for improving the characteristics of a mineral lubricating oil, may be solvent extracted with a liquid material which has selective solvent power for only a part of the reaction product. Comparable improved additives (extract) are obtained thereby.

In view of the foregoing disclosures, variations and modifications will be apparent to those skilled in the art. The invention contemplates all such variations and modifications except as do not come within the appended claims.

We claim:

1. A method of treating the reaction product of an organic compound and a phosphorus sulfide which contains at least one material selected from the group consisting of material impairing its characteristics as an oxidation-inhibiting additive for an acid-refined mineral lubricating oil and material impairing its characteristics as a corrosion-inhibiting additive for a solvent-extracted mineral lubricating oil, to remove said impairing materials therefrom, which comprises mixing said reaction product as a substrate with a liquid material having a selective solvent power for a part of said substrate, with formation of an extract phase and a raffinate phase, separating the extract phase from the raffinate phase and recovering from the extract phase a superior mineral lubricating oil additive having improved inhibiting properties consisting of the extract.

2. A method in accordance with claim 1 in which the liquid material having a selective solvent power for a part of said substrate is a nonpolar material.

3. A method in accordance with claim 2 in which the nonpolar material is a hydrocarbon.

4. A method in accordance with claim 1 in which the reaction product is of an olefin and a phosphorus sulfide.

5. A method in accordance with claim 4 in which the phosphorus sulfide is phosphorus pentasulfide.

6. A method in accordance with claim 1 in which the reaction product is of degras and phosphorus pentasulfide and the liquid material is acetone.

7. A method in accordance with claim 1 in which the reaction product is of motor polymer and phosphorus pentasulfide and the liquid material is a hydrocarbon having not over 8 carbon atoms per molecule.

8. A method in accordance with claim 1 in which the reaction product is of reduced motor polymer and phosphorus pentasulfide and the liquid material is liquid propane.

9. A method in accordance with claim 1 in which the reaction product is of reduced motor polymer and phosphorus pentasulfide and the liquid material is n-pentane.

10. A method in accordance with claim 1 which includes forming a metal derivative of said extract.

11. A method in accordance with claim 10 in which the liquid material is a nonpolar material.

12. A method in accordance with claim 10 in which the liquid material is an oxygenated material.

13. A method of treating the reaction product

of an organic compound and a phosphorus sulfide to remove therefrom material contained therein impairing its characteristics as a corrosion-inhibiting additive for a solvent-extracted mineral lubricating oil, which comprises mixing said reaction product as a substrate with a liquid material having a selective solvent power for a part of said substrate, with formation of an extract phase and a raffinate phase, separating the extract phase from the raffinate phase and recovering from the extract phase a superior mineral lubricating oil additive consisting of the extract.

14. A method of treating the reaction product of an organic compound and a phosphorus sulfide to remove therefrom material contained therein impairing its characteristics as an oxidation-inhibiting additive for an acid-refined mineral lubricating oil, which comprises mixing said reaction product as a substrate with a liquid material having a selective solvent power for a part of said substrate, with formation of an extract

phase and a raffinate phase, separating the extract phase from the raffinate phase and recovering from the extract phase a superior mineral lubricating oil additive consisting of the extract.

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