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EXTREME PRESSURE LUBRICANT

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This invention deals essentially with extreme pressure lubricants adapted for carrying much higher loads than can be carried safely by the usual unblended lubricants. More specifically, it involves the use of certain phosphorus, sulfur and halogen containing compounds for improving the load carrying capacity of ordinary lubricants such as mineral lubricating oils.

A number of phosphorus compounds and derivatives thereof have been suggested in the art for use as extreme pressure lubricating agents. These products, however, involve the formation of considerable amounts of sludge during their manufacture or, if prepared from phosphorus halides, they may contain free hydrochloric acid which is corrosive to steel. Most of them also possess only weak pressure-resisting properties, thereby involving danger of scoring of the hypoid gears or other moving parts lubricated thereby.

One purpose of this invention is to avoid these prior art difficulties by producing a substantially non-corrosive extreme-pressure agent without excessive sludge formation capable of carrying high loads and retaining its stability over a long period of time.

According to the invention, a halogenated aliphatic, aromatic or terpene compound, especially one of at least 8–10 carbon atoms, e. g. a chlorinated hydrocarbon such as chlorinated paraffin or petrolatum wax, or a halogenated fatty acid or ester, e. g. chlorstearic acid or chlorethyl stearate, or chlorinated alcohol or ether, or similar compound containing at least 5 or 10% chlorine is reacted with 1–10% phosphorus sesquisulfide (P_4S_3), at about 300–400° F. until reaction is substantially complete. Other phosphorus sulfides are not as effective as P_4S_3 since they tend to produce excessive sludging and do not give products as rich in phosphorus and sulfur. The product thus prepared may be neutralized if desired to remove any excess halogen halide liberated by the reaction, and then blended in the lubricating base stock in amounts of 1 to 10%, more or less.

In the preferred embodiment, chlorinated paraffin wax containing about 30% Cl is heated to 300–400° F. with 2–5% P_4S_3 , producing a reaction product containing 27% Cl, 1.5% S, and 1.0% P. This may then be blended in 5% concentration with 85% untreated mineral oil and 10% sulfurized fatty oil, especially a non-corrosive sulfurized oil of the type of sperm oil or sulfurized fatty alcohols, such as cetylalcohol, degeras alcohols (e. g. wool fat) to produce a highly suitable extreme-pressure lubricant for hypoid lubrica-

tion, production of cutting oils (soluble or non-aqueous), and in general, for uses requiring a lubricant capable of withstanding high load and pressures. Another raw material for the preparation of the blending agent previously described is the by-product overhead "slop" cut from "Paraflow" manufacture. This material, which essentially contains unreacted wax, naphthalene, alkylated naphthalenes, etc., is chlorinated to the extent of about 20 or 30% Cl before it is reacted with the P_4S_3 .

It is essential that a halogenated compound be reacted with the P_4S_3 , since ordinary mineral or fatty oils or other compounds devoid of halogen, outside of giving excessive sludging, show very little reaction with P_4S_3 to give a satisfactory yield of an oil soluble phosphorus and sulfur-containing product of satisfactory E. P. properties. The products produced according to the present invention have also been found suitable as lubricant anti-oxidants and bearing corrosion inhibitors when employed in small amounts, in the neighborhood of 0.1%.

An alternative method which is also satisfactory but not convenient as the one just outlined, is to react a fatty or mineral oil or synthetic oil simultaneously with chlorine and a sulfide of phosphorus, e. g. P_4S_3 , P_2S_5 , P_2S_3 , $PSCl_3$, or mixtures of phosphorus and sulfur.

If the chlorinated compound is treated with 3% or less of the phosphorus sulfide in a finely divided state at 250 to 370° F., a satisfactory product is obtained. Excessive heating, especially at higher temperatures, should be avoided, since it is conducive to coking and formation of a less active and stable product. With larger amounts of phosphorus sulfide, higher temperatures and longer periods of treating are necessary. At about 250° F., copious white fumes are liberated, indicating that reaction temperature is reached, and as the heating progresses, some glowing may occur. After the material has cooled, the product containing chlorine, sulfur and phosphorus is dissolved in ordinary lubricating oils, with or without the addition of other blending agents.

The following examples illustrate some of the many phases involved in this invention:

Example 1

A Paraflow "slop" wax is chlorinated to about 30% chlorine and treated with phosphorus sesquisulfide as outlined above. The product is then dissolved in 10% concentration to a Pennsylvania gear lubricating oil of 200 seconds Saybolt

viscosity at 200° F. The resulting blend carried a load of 15,000 lbs. in the Almen test, with low friction (35) and perfect pin condition. In the S. A. E. machine test, the blend carried a load corresponding to 390 lbs. scale reading at 1000 R. P. M. with a 14.6:1 loading ratio. In the copper strip corrosion test, the blend showed only a slight bronzing of the copper strip after 3 hrs. immersion at 210° F.

Example 2

Two parts of P_4S_3 reacted with 20 parts of chlorinated "slop" wax (containing 30% chlorine) are dissolved in 78 parts of a gear mineral lubricating oil of about 140 seconds Saybolt viscosity at 200° F. In the S. A. E. machine test, the blend carried a load corresponding to a 580 lbs. scale reading at 1000 R. P. M. and 14.6:1 loading ratio. Only a slight bronzing of the copper strip in the copper strip test was encountered after 3 hrs. at 210° F.

Example 3

About 10% of P_4S_3 is added to sperm oil in a dispersion. While mixing the suspension, chlorine gas is passed through the suspension, and a rise in temperature of 100–150° F. occurs immediately. After the reaction mixture becomes clear, a stream of air is blown through the product to remove any free HCl. After air blowing, the product is dissolved in a lubricating oil in 5% concentration, and this blend passes the Almen test of 15 weights (gradual loading), and 13 weights (shock loading), with excellent pin condition and low frictional readings.

During the introduction of the chlorine through the P_4S_3 suspension in sperm oil, some PCl_3 is formed which condenses on the sides of the flask. This material may be removed and replaced in the sperm oil, if desired. No sludge is formed during this reaction if P_4S_3 is employed, although this is not the case if P_2S_3 or P_2S_5 or other phosphorus compounds are cooked into fatty or mineral oils.

Example 4

About 98 parts of 118–120° F. melting point paraffin wax is chlorinated to about 40% chlorine content and reacted with 2 parts of P_4S_3 at 370° F. maximum temperature. The reaction product of the chlorinated wax and P_4S_3 is then blended with mineral oil and a non-corrosive sulfurized sperm oil, the final blend having the following composition:

	Per cent
P_4S_3 treated chlorinated wax.....	7
Non-corrosive sulfurized sperm oil.....	10
Paraffinic or naphthenic base mineral gear oil (90 seconds viscosity at 210° F.).....	83

This blend imparts a very slight bronzing to a copper strip in the copper strip corrosion test at 210° F. On the S. A. E. machine test it carries a load of 320–340 lbs. scale reading at 1000 R. P. M. and 14.6:1 loading ratio.

Example 5

Seven parts of chlorinated wax having a 40% chlorine content are blended with 10 parts of a non-corrosive sulfurized sperm oil. This mixture is then reacted at 250–370° F. with 0.15 part of P_4S_3 . This clear, dark red product is compounded with 83 parts of mineral gear oil to give a clear, non-sludging extreme-pressure lubricant. A lubricant prepared by this method, carried 15 weights gradual and shockloading on 70

the Almen machine test at 600 R. P. M. with excellent pin condition, and on the S. A. E. machine test carried a load corresponding to 340 lbs. scale reading at 1000 R. P. M. and 14.6:1 loading ratio. On the copper strip corrosion test (3 hrs. at 210° F.) only a slight bronzing of the copper strip was encountered.

Example 6

About 98 parts of melted 118–120° F. melting point paraffin wax are mixed with 2 parts of P_4S_3 . In a suitable apparatus, chlorine gas is passed through the mixture at 150–200° F. until the mixture has been chlorinated to approximately 40% chlorine content. This material is air blown to remove any entrapped hydrochloric acid gas. After air blowing until no appreciable sharp odor is noticed in the product, the clear, yellow, syrupy product is blended with a non-corrosive sulfurized fatty oil and mineral oil to give the following composition:

	Per cent
The above reaction product.....	7
Non-corrosive sperm fatty oil.....	10
Mineral gear oil (90 vis. Saybolt at 210° F.)..	83

This blend, when tested on the S. A. E. machine test, carried a load corresponding to 325/350 scale pull at 1000 R. P. M. and 14.6:1 loading ratio. On the copper strip corrosion test (heating 3 hrs. at 210° F.) and the General Motors steel corrosion test (heating 75 parts of the lubricant and 25 parts of water for 18 hours at 200° F. in contact with a steel rod) only a very slight bronzing of the copper and no discoloration of the steel rod was effected.

Other materials such as polymer thickeners or V. I. improvers, soluble metal soaps (e. g. aluminum, zinc or lead naphthenate, calcium stearate or phenyl stearate, etc.), anti-oxidants, dyes, pour inhibitors, resins, volatilized mineral and/or fatty oils, fatty or mineral residua, or pitches, phosphorus esters, oiliness agents, sulfurized terpenes, sulfurized mineral oils and extracts, sulfur compounds occurring in petroleum or synthetically prepared sulfur compounds, especially those having a low volatility and freedom of odor, and the like, may be added to these compositions.

The compounds produced according to this invention may also be added to greases, industrial oils and lubricants, Diesel fuels, slushing oils, and the like.

The reaction product per se made according to this invention, and the process of preparing same are claimed in copending application Serial No. 412,596, filed September 27, 1941.

We claim:

1. An extreme pressure lubricant comprising a mineral lubricating oil, an oil sulfurized fatty oil, and a soluble reaction product of phosphorus sesquisulfide with a halogenated aliphatic hydrocarbon compounds containing at least 5% halogen.

2. An extreme pressure lubricant according to claim 1 in which the proportions of the individual ingredients is approximately 85% mineral lubricating oil, 10% sulfurized fatty oil and 5% reaction product.

3. An extreme pressure lubricant comprising a mineral lubricating oil and a small but effective amount of an oil-soluble reaction product of a phosphorus sulfide with a halogenated hydrocarbon compound containing at least 5% halogen.

4. An extreme pressure lubricant according to

claim 3 in which the reaction product contains approximately 27% Cl, 1.5% S, and 1% P.

5. An extreme pressure lubricant comprising a mineral lubricating oil and a small amount of an oil-soluble organic compound serving as an extreme pressure lubricating agent and containing halogen, sulfur and phosphorus, said agent being a reaction product of a phosphorus sulfide with a halogenated hydrocarbon compound.

6. An extreme pressure lubricant comprising a major proportion of a mineral lubricating oil and a minor proportion of an oil-soluble reaction product of phosphorus sesquisulfide with a halogenated aliphatic hydrocarbon compound containing at least 5% halogen.

7. An extreme pressure lubricant comprising a major proportion of a mineral lubricating oil and about 1 to 20% of a non-corrosive, stable extreme pressure lubricating agent made by treating a chlorinated aliphatic hydrocarbon compound having at least 8 carbon atoms and containing at least 5% chlorine with about 1 to 10% of phosphorus sesquisulfide at a temperature of about 250 to 400° F.

8. An extreme pressure lubricant comprising a major proportion of a mineral lubricating oil and about 1 to 10% of a non-corrosive, stable extreme pressure lubricating agent made by treating chlorinated paraffin wax containing about 30 to 40% chlorine with about 2 to 5% of phosphorus sesquisulfide at about 250 to 400° F.

9. An extreme pressure lubricant comprising a mineral lubricating oil and an extreme pressure lubricating agent containing halogen, sulfur and phosphorus, said sulfur and phosphorus being derived solely from phosphorus sesquisulfide and said agent being an oil-soluble reaction product of phosphorus sesquisulfide with a halogenated aliphatic hydrocarbon containing at least 5% halogen.

10. An extreme pressure lubricant comprising a major proportion of a mineral lubricating oil and about 1 to 20% of a substantially non-corrosive, stable extreme pressure lubricating agent containing about 27% chlorine, about 1.5% sulfur and about 1% phosphorus, said agent being made by treating a chlorinated aliphatic hydrocarbon compound containing at least 10% halogen with phosphorus sesquisulfide.

11. An extreme pressure lubricant comprising a major proportion of a mineral lubricating oil and a small amount of an extreme pressure lubricating agent containing halogen, sulfur, and phosphorus, said agent being made by treating an aliphatic organic compound with phosphorus sesquisulfide with simultaneous treatment with chlorine.

12. An extreme pressure lubricant comprising a major proportion of a mineral lubricating oil and about 1 to 20% of an extreme pressure lubricating agent made by the process which consists in chlorinating an aliphatic hydrocarbon material having at least 8 carbon atoms, to a chlorine content of at least 5%, and reacting the resulting chlorinated aliphatic hydrocarbon with about 1% to 10% of a phosphorus sulfide at a temperature of about 250° to 400° F.

13. An extreme pressure lubricant comprising a major proportion of mineral lubricating oil and about 1% to 20% of a substantially non-corrosive oil-soluble stable extreme pressure lubricating agent made by the process which consists in chlorinating a petroleum wax to a chlorine content of about 30% to 40%, and reacting the resultant chlorinated wax with about 2% to 5% of phosphorus sesquisulfide at a temperature of about 250° to 400° F.

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CERTIFICATE OF CORRECTION.

Patent No. 2,307,183.

January 5, 1943.

JOHN C. ZIMMER, ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, second column, line 59, claim 1, strike out "an oil" and insert --a--; line 60, strike out "a" and insert --an oil--; page 3, first column, line 8, for "rection" read --reaction--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 30th day of March, A. D. 1943.

Henry Van Arsedale,

Acting Commissioner of Patents.

(Seal)

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