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# 2,836,561
# LUBRICATING OILS

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7 Claims. (Cl. 252—33.6)

This invention is for improvements in or relating to lubricating oils, and has particular reference to lubricating oils having extreme pressure properties, and to cutting oils and metal working oils.

A large number of organic compounds have been proposed for use as extreme pressure agents, including various types of organic di- and polysulphides, and the corresponding derivatives of selenium. While the higher polysulphides are very effective in increasing the film rupture strength of the oil in which they are dissolved, they tend to be inherently unstable, corrosive, especially to copper and cuprous materials, and to exert a pro-oxidant effect upon the oil.

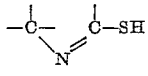
The potential corrosiveness of a sulphur-bearing organic compound towards cuprous metals is dependent both on the chemical structure of the compound and on the temperature at which it is employed. Thus, in general, monosulphides are less corrosive than disulphides which, in turn, are less corrosive than the tri- or higher polysulphides; aromatic polysulphides, in which the sulphur is directly linked to aromatic nuclei, are less corrosive than the corresponding aliphatic compounds, while the corrosiveness of any particular sulphide group can be appreciably influenced by the presence of other neighbouring groups.

Generally speaking the effectiveness of sulphur-bearing organic compounds as extreme pressure additives for lubricating oils depends upon the ease with which they break down, yielding sulphur which then reacts with the metal forming a sulphide film which prevents welding at high loads. Thus, for example, the tri- and tetrasulphides will provide reactive sulphur at lower temperatures than the corresponding disulphides, and consequently are more effective extreme pressure additives. Their corrosiveness to copper and cuprous metals, however, is a serious drawback, precluding their use in certain circumstances.

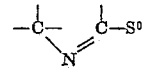
It is an object of the present invention to provide extreme pressure lubricants containing organic di- or polysulphides, or their selenium analogues, which are normally corrosive to copper at 300° F. or below, in which the corrosiveness to copper or cuprous alloys is inhibited by the addition of a novel combination of inhibitors.

It has already been proposed in United States patent specification No. 2,414,257 to inhibit the copper staining tendencies of lubricants containing "active" sulphur by the addition of a minor proportion of a mercapto-arylene thiazole or a derivative thereof, or an alkyl thiuram mono- or disulphide.

We have now found that the staining of copper and cuprous metals by lubricants containing organic di- and polysulphides or their selenium analogues can be inhibited to an outstanding and unexpected degree by the conjoint use of two additives of different types. These are: (*a*) a cyclic compound containing the group



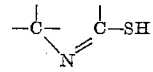
as part of a ring structure or a derivative thereof capable of homolytic dissociation into free radicals



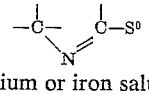


and (*b*) a zinc, cadmium or iron salt of an organic dithiocarbamic acid.

According to the present invention there is provided a lubricating composition comprising a lubricant base and a minor proportion, sufficient to impart extreme-pressure properties to the lubricant, of an organic di- or polysulphide or their selenium analogues, together with a minor proportion of (*a*) a cyclic compound containing the group

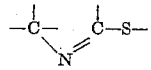


as part of a ring structure or a derivative thereof capable of homolytic dissociation into free radicals



and (*b*) a zinc, cadmium or iron salt of an organic dithiocarbamic acid.

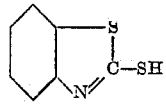
More specifically additive (*a*) is a compound of general formula A—B in which A is a radical containing the group



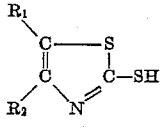
and B is a radical attached to the sulphur atom, of such nature that the compound A—B dissociates homolytically at elevated temperatures yielding free radicals. Thus, B may be H, —$CH_2OH$, —$C(C_6H_5)_3$, or it may be the same as A.

We have found that if B is a group, such as benzyl, butylaceto or dioctyl ammonium, which encourages heterolytic dissociation, the compound is relatively ineffective as an inhibitor of copper staining.

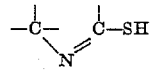
A preferred sub-class of inhibitors of this type are 2-mercaptobenzothiazole



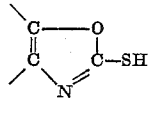
and its derivatives such as benzothiazole disulphide, hydroxymethyl thiobenzothiazole and triphenyl methyl thiobenzothiazole, but the corresponding derivatives of other 2-mercaptothiazoles may be employed, for example, those having the general formula



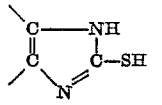
where $R_1$ and $R_2$ may be the same or different and represent hydrogen or hydrocarbon radicals, e. g., 2-mercapto-4-methyl-5-isopropyl thiazole. Examples of other types of additive containing the group



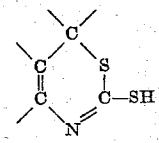
as part of a ring structure are the 2-mercapto-oxazoles containing the group



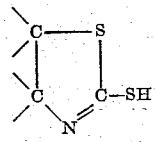
the 2-mercaptoimidazoles containing the group



the 2-mercaptothiazines containing the group



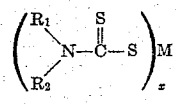
or isomers thereof and the 2-mercaptothiazolines containing the group



It is to be understood that the foregoing examples are merely illustrative of the more readily available types of compounds falling within category (*a*), and the invention is not intended to be restricted to these types. It is also to be understood that derivatives of these mercapto compounds may be employed, which dissociate homolytically yielding free radicals, e. g., the disulphides.

The substituents carried by the carbon atoms of the above ring systems are normally H, alkyl or aryl, e. g., benzo radicals, although other types of substituents, e. g., alkylamino, may be present if desired. It is necessary to select the substituents having regard to the nature of the heterocyclic ring, so that the resulting compound is soluble in the lubricant in the proportion in which it is desired to employ it. Thus, 2-mercaptobenzothiazole has a limited though adequate solubility in most mineral lubricating oils, whereas other closely related compounds, e. g., 2-mercapto-4-methyl thiazole, 2-mercaptobenzoxazole and 2-mercaptobenzimidazole, have not. The latter two compounds, however, may be used in certain synthetic lubricants, e. g., di(2-ethyl hexyl) sebacate, if desired. Mineral oil-soluble compounds containing oxazole and imidazole rings can, however, be obtained by choosing suitable alkyl or alkylated aromatic substituents. The most effective compounds appear to be those containing a benzo radical adjacent to the heterocyclic nucleus.

Additive (*b*) is a metal salt of an organic dithiocarbamic acid having the general formula



where $R_1$ and $R_2$ may be the same or different and represent preferably hydrocarbon radicals although either $R_1$ or $R_2$ may be H, or may together form part of a ring structure, M is zinc, cadmium or iron, and $x$ is the valency of M.

Specific examples of additives of this type are:

Zinc diethyl dithiocarbamate
Zinc di-n-butyl dithiocarbamate
Zinc di (methyl cyclopentamethylene) dithiocarbamate
Cadmium di n-butyl dithiocarbamate
Ferric di n-butyl dithiocarbamate
Zinc mono-octyl dithiocarbamate
Cadmium pentamethylene dithiocarbamate

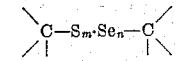
Whereas the inhibitors of group (*a*) are in themselves effective inhibitors of copper staining by "active" sulphur, the compounds of group (*b*) are of little or no value in this respect, although their use in lubricating oils as inhibitors of oxidation has been known for some time.

The conjoint use of additives of these two types, however, inhibits the staining of copper and cuprous metals by organic di- and polysulphides and their selenium analogues to an extent not obtainable by the use of the inhibitors of group (*a*) alone, thus enabling any given lubricant to be used at higher temperatures than would otherwise be possible. Furthermore, it has been found that the more effective inhibitors of group (*a*) when used alone protect the metal from sulphur attack by forming



on the surface a coarse yellowish-brown film of the copper complex which is capable of being removed by rubbing with consequent loss in weight of the metal. By the conjoint use of the two types of additive, however, it is often possible to obtain a bright untarnished copper surface.

The extreme pressure additives employed in the lubricating compositions of the present invention contain the group



where $m$ is 0, 1, 2, 3 or 4 and $n$ is 0, 1, 2 or 3 the sum of $m$ and $n$ being not less than 2 and not greater than 4, the carbon atoms being attached to other carbon atoms or to hydrogen or halogen atoms.

More specifically these compounds may be simple organic di, tri or tetrasulphides, di or tri-selenides or sulphoselenides. They may be aliphatic, aromatic or alicyclic compounds which may be unsubstituted or substituted by e. g., halogen atoms, nitro groups, hydroxyl groups or ester groups. Alternatively, they may be cyclic compounds in which the sulphur and/or selenium-containing groups form part of a ring structure.

Alternatively, the extreme pressure additives may be complex compounds or mixtures of compounds obtained, for example, by sulphurising a wide variety of unsaturated organic compounds such as lard oil, sperm oil, oleine, tallow, wool fat or various terpene derivatives. The precise chemical structure of these compounds is not known with certainty. It is known, however, that when one or less than one molecular proportion of sulphur per olefinic linkage is employed in sulphurising an unsaturated organic compound, the product is generally non-corrosive towards copper at 300° F. or higher; it is believed that in such materials the sulphur is present as mono-sulphide linkages. When higher amounts of sulphur are used, however, products are obtained having varying degrees of chemical activity towards copper, depending upon the amount of sulphur used and tempertaure of sulphurizing. It is believed in these latter compounds di- and polysulphide linkages exist which may form bridges between the molecules or be present in cyclic structures.

The amounts of the extreme pressure additives employed will depend upon the purpose for which the oil is to be used. Any quantity from the minimum sufficient to impart a substantial increase in film rupture strength up to about 10 percent may be employed, but in general from about 0.5 percent to about 2.0 percent is contemplated, especially for use in turbine oils.

Specific examples of the extreme pressure additives employed in the present invention are:

Dibenzyl disulphide
Dibenzyl trisulphide
Di(3-carbomethoxy-4-hydroxyphenyl) polysulphides as described in United States patent specification No. 2,388,047
Sulphurized sperm oil
Di(n-butyl) dithiodiacetate
Di(n-butyl) trithiodiacetate
Di n-butyl tetrathiodiacetate
Di n-butyl diselenodiacetate
4.7 - dimethyl - 4.7 - di(2'dimethyl propyl) - 1,2,5,6-tetrathiacyclo-octane

Halogenated derivatives of the foregoing may also be employed.

It will be understood that the present invention applies not only to lubricants containing a di- or polysulphide or selenium analogue as the sole extreme pressure additive, but also to lubricants containing additionally other additives known to confer extreme pressure properties such as organic halogenated compounds and organic phosphorus compounds. A large number of such compounds are known to the art.

In one application of the present invention an extreme

pressure lubricant for the lubrication of hypoid gears comprises a mineral lubricating oil, an organic di- or polysulphide compound (or selenium analogue) of the type hereinbefore described, and an organic halogenated compound e. g., a chlorinated paraffin wax, together with the two additives of types (*a*) and (*b*) to inhibit staining of copper and cuprous metals.

In another application of the invention a turbine oil having extreme pressure properties comprises a mineral lubricating oil and an extreme pressure additive of the type described in United States patent specification No. 2,691,000 having the general formula:

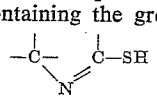
$$ROOC.CH_2—(X)_n—CH_2.COOR$$

where R is an alkyl, aryl or cycloalkyl radical, X is sulphur or selenium and *n* is 2, 3 or 4 when X is sulphur, and 2 or 3 when X is selenium, together with additives of types (*a*) and (*b*) hereinbefore described, to inhibit staining of copper and cuprous metals. Mixed sulphoselenides conforming to the above general formula are included within the scope of the invention.

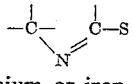
While the present invention is particularly concerned with lubricating compositions having a mineral lubricating oil base, it is also applicable to lubricants consisting wholly or predominantly of fatty oils, e. g., castor oil or rapeseed oil, and to synthetic lubricants such as dicarboxylic acid diesters, e. g., di(2-ethyl hexyl) sebacate, and polyglycol ethers.

In carrying out the present invention, the organic di- or polysulphide extreme pressure additive is present in sufficient quantity to impart extreme pressure properties to the oil while the inhibitors may each be present in amounts ranging from about 0.01 to about 1.0 percent. In general the best results are obtained by the use of the two inhibitors in approximately equal amounts.

The present invention also includes an additive for use in a lubricating composition, containing as an extreme-pressure additive an organic di- or polysulphide or their selenium analogues, to inhibit the copper or cuprous metal staining properties of the extreme-pressure additive, which additive comprises a mixture of (*a*) a cyclic compound containing the group



as part of a ring structure or a derivative thereof capable of homolytic dissociation into free radicals



and (*b*) a zinc, cadmium or iron salt of an organic dithiocarbamic acid.

The following examples are illustrative of compositions prepared in accordance with the present invention:

## EXAMPLE I

An extreme pressure hypoid rear axle lubricant, conforming to the Society of Automotive Engineers (S. A. E.) classification grade 90, was prepared having the following approximate composition:

75% conventionally refined Mid-Continent oil of viscosity about 700 seconds Redwood at 140° F.
15.91% solvent refined mineral oil of viscosity 150 seconds Redwood at 140° F.
8% chlorinated paraffin wax (approx. 40% chlorine)
0.5% di(3-carbomethoxy-4-hydroxyphenyl) polysulphides prepared as described in Example 4 of United States patent specification No. 2,388,047
0.25% di(3-carbomethoxy-4-hydroxyphenyl) thioether
0.14% 2-mercaptobenzothiazole
0.20% zinc di-n-butyl dithiocarbamate

## EXAMPLE II

A synthetic lubricant having extreme pressure properties consisting of di(2-ethyl hexyl) sebacate, having dissolved therein:

1.0% di(3-carbomethoxy-4-hydroxyphenyl) disulphide (prepared as described in Example 2 of United States patent specification No. 2,388,047
0.1% benzothiazole disulphide
0.1% zinc diethyl dithiocarbamate

## EXAMPLE III

An extreme pressure turbine oil conforming to British Admiralty specification O. M. 100 was prepared, consisting of approximately:

75% of a mineral oil having a viscosity of about 170 seconds Redwood at 140° F.
25% of a solvent refined mineral oil having a viscosity of about 65 seconds Redwood at 140° F.

to which blend was added:

1.5% di-n-butyl trithiodiacetate
0.1% benzothiazole disulphide
0.1% zinc di-n-butyl dithiocarbamate
0.07% ethylene glycol mononaphthenate acid phthalate (ferrous metal corrosion inhibitor)
0.001% calcium petroleum sulphonate

## EXAMPLE IV

A cutting oil was prepared consisting of:

40% conventionally refined naphthenic base oil of viscosity about 140 seconds Redwood at 140° F.
45% mineral oil of viscosity about 50 seconds Redwood at 140° F.
10% sulphurised olein
5% chlorinated paraffin wax (approx. 40% chlorine)

to which blend was added:

0.1% 2-mercaptobenzothiazole
0.1% zinc di-n-butyl dithiocarbamate

The sulphurised olein used in this example contained about 24% sulphur and was prepared by heating olein with sulphur at a temperature of about 170° to 200° C. for a period of about 40 minutes and then heating the product with further sulphur at a slightly lower temperature, e. g. 160° to 170° C.

*Table 1*

| Test No. | Inhibitor (*a*) | Percent | Percentage of inhibitor (*b*) (zinc di-n-butyl dithiocarbamate) | Appearance of copper strips after— | |
|---|---|---|---|---|---|
| | | | | 3 hours | 24 hours |
| 1 | None | | None | Smooth black | Black, much flaking of copper sulphide. |
| 2 | do | | 0.2 | do | Black, considerable flaking. |
| 3 | Benzothiazole disulphide | 0.2 | None | Dull copper | Dark brown, with surface film. |
| 4 | do | 0.1 | 0.1 | Bright copper | Light peacock stain. |
| 5 | Hydroxymethylthiobenzothiazole. | 0.2 | None | Light brown | As No. 3. |
| 6 | do | 0.1 | 0.1 | Light peacock | Medium brown/peacock. |
| 7 | 2-Mercapto-4,6,6 trimethyl-1,3,6 H thiazine. | 0.2 | None | Purple brown | Black, considerable flaking. |
| 8 | do | 0.1 | 0.1 | Light brown/peacock. | Light brown with some dark patches. |

Test results in this table illustrate the effect of adding zinc di-n-butyl dithiocarbamate to a lubricating oil consisting of the same mineral oil blend as in Example III, containing 1.5% di-n-butyl trithiodiacetate, to which various copper stain inhibitors of type (*a*) had been added.

The tests were carried out by immersing clean copper strips, polished with carborundum powder, in the oils contained in 1" test-tubes. The test-tubes were placed in an oil bath which was maintained at the desired temperature in an oven. The oils were heated at 100° C. for 24 hours.

*Table 2*

| Test No. | Inhibitor (*a*) | Percent | Percentage of inhibitor (*b*) (zinc di-n-butyl dithiocarbamate) | Appearance of copper strips after— | |
|---|---|---|---|---|---|
| | | | | 3 hours | 24 hours |
| 9 | 2-Mercapto-benzoxazole | 0.2 | None | Patches of black, brown and peacock. | Smooth black. |
| 10 | do | 0.1 | 0.1 | Bright, brassy with a little light peacock stain. | Light peacock. |
| 11 | 2-Mercapto-benzimidazole | 0.2 | None | Brown with black patches | As No. 9. |
| 12 | do | 0.1 | 0.1 | As No. 10 | As No. 10. |

In this table a typical synthetic lubricant, di(2-ethyl hexyl) sebacate was employed, containing 1.5% di-n-butyl trithiodiacetate, the temperature being 100° C. as before.

*Table 3*

| Test No. | Percentage of benzothiazole disulphide | Inhibitor (*b*) | Percent | Appearance of copper after— | |
|---|---|---|---|---|---|
| | | | | 3 hours | 24 hours |
| 1 | None | None | | Smooth black | Black, much flaking of copper sulphide. |
| 3 | 0.2 | do | | Dull copper | Dark brown with surface film. |
| 13 | 0.1 | do | | Light brown | Dark brown, dull with small black patches. |
| 14 | 0.1 | Zinc dimethyl dithiocarbamate. | 0.1 | Bright copper | Bright copper. |
| 15 | 0.1 | Zinc diethyl dithiocarbamate. | 0.1 | do | Light brown with patches of bright copper. |
| 16 | 0.1 | Cadmium dibutyl dithiocarbamate. | 0.1 | do | Almost bright copper. |
| 17 | 0.1 | Cadmium pentamethylene dithiocarbamate. | 0.1 | do | Brown/peacock. |
| 18 | 0.1 | Ferric dibutyl dithiocarbamate. | 0.2 | do | Bright orange. |
| 19 | 0.1 | Nickel dibutyl dithiocarbamate. | 0.2 | Dull copper | Very dark brown with surface film. |
| 20 | 0.1 | Piperidine cyclopentamethylene dithiocarbamate. | 0.1 | Dark brown, black patches. | Dark brown, black flaky patches. |

The results in Table 3 illustrate the effect of adding various dithiocarbamates to a mineral oil blend as in Table 1, containing 1.5% di-n-butyl trithiodiacetate and 0.1% of benzothiazole disulphide. The test temperature was 100° C. as before.

When the foregoing dithiocarbamates were added in 0.2% concentration to the oil in the absence of the benzothiazole disulphide, no appreciable improvement was observed, the copper strips being blackened after 3 hours.

The last two tests in this table illustrate the ineffectiveness of certain dithiocarbamates closely related to those of the present invention. A considerable number of dithiocarbamates of different metals have been tested, but only those of zinc, cadmium and iron proved to be effective.

*Table 4*

| Test No. | Sulphur or selenium compound | Percent | Percent of benzothiazole disulphide | Percent of zinc dibutyl dithiocarbamate | Appearance of copper after— | |
|---|---|---|---|---|---|---|
| | | | | | 3 hours | 24 hours |
| 21 | Dibenzyl trisulphide | 1.5 | None | None | Black, flaky | Black, much flaking. |
| 22 | do | 1.5 | 0.2 | None | Light peacock | Do. |
| 23 | do | 1.5 | None | 0.2 | Smooth black | Do. |
| 24 | do | 1.5 | 0.1 | 0.1 | Bright copper | Smooth black. |
| 25 | Di-n-butyl diseleno-diacetate. | 1.0 | None | None | Dark brown | Black, slightly flaky. |
| 26 | do | 1.0 | 0.2 | None | Greenish brown | Dark brown, patchy. |
| 27 | do | 1.0 | None | 0.2 | Peacock | Dark peacock. |
| 28 | do | 1.0 | 0.1 | 0.1 | Bright copper | Bright copper with brown patches. |
| 29 | 4.7 dimethyl-4,7 di (2'dimethyl propyl)-1,2,5,6 tetrathia-cyclo-octane. | 1.0 | None | None | Very dark brown | Black, flaky. |
| 30 | do | 1.0 | 0.2 | None | Very light peacock | Do. |
| 31 | do | 1.0 | None | 0.2 | Brown | Do. |
| 32 | do | 1.0 | 0.1 | 0.1 | Bright copper | Smooth black. |

The results in Table 4 illustrate the variety of organic di- and polysulphides and their selenium analogues which may be inhibited by the combination of additives of the present invention.

The compounds were dissolved in the same mineral oil blend as used in Table 1, the test being of 24 hours' duration at 100° C.

The compound used in tests 29 to 32 was prepared by treating diisobutylene (2 mols) in benzene solution with sulphur monochloride (1 mol) and reacting the product with alcoholic sodium disulphide to yield a product containing about 30% sulphur, consisting principally of the above compound.

*Table 5*

| Test No. | Sulphur compound | Percent | Percent of 2-mercaptobenzothiazole | Percent of zinc dibutyl dithiocarbamate | Appearance of copper after 18 hours |
|---|---|---|---|---|---|
| 33 | Di-tertiary butyl disulphide | 2.0 | None | None | Black, very flaky. |
| 34 | do | 2.0 | 0.2 | None | Dark brown-black flaky patches. |
| 35 | do | 2.0 | 0.1 | 0.1 | Orange with dull deposit. |
| 36 | Sulphurized sperm oil | 5.0 | None | None | Very dark brown, flaky. |
| 37 | do | 5.0 | 0.2 | None | Do. |
| 38 | do | 5.0 | None | 0.2 | Do. |
| 39 | do | 5.0 | 0.1 | 0.1 | Brown/peacock, slightly flaky. |

Table 5 sets out the results of further tests which were carried out in the same base oil blend, but for 18 hours at 275° F.

*Table 6*

| Test No. | Oil Composition | Appearance of copper after— | |
|---|---|---|---|
| | | 3 hours | 24 hours |
| 40 | Composition of Example I | Bright gold | Golden. |
| 41 | Composition of Example I without the zinc di-n-butyl dithiocarbamate. | Peacock, with dark spots. | Brown/peacock with black spots. |
| 42 | Composition of Example IV | Bright copper with with dark spots. | Dark peacock—smooth black in places. |
| 43 | Composition of Example IV without the zinc di-n-butyl dithiocarbamate, but with an extra 0.1% of 2-mercaptobenzothiazole. | Dark peacock | Black, flaky. |

It will be understood that the foregoing tests are designed to demonstrate the superiority of the mixed additives of types (*a*) and (*b*) as compared with either type used alone. At higher temperatures, or in the presence of more active sulphur compounds, it may be necessary to use more than the 0.1–0.2 percent of the additives quoted in the above examples, provided that compounds are selected having adequate solubility in the lubricating oil base. On the other hand, in other circumstances, e. g., to inhibit staining of copper at room temperature, quite small amounts of inhibitors, below 0.1 percent, may be sufficient.

We claim:

1. A turbine oil comprising mineral oil containing from 0.5 to 2.0 percent of a compound selected from the group consisting of di(n-butyl) dithiodiacetate and di(n-butyl) trithiodiacetate, together with from 0.05 to 0.2 percent of benzothiazole disulphide and from 0.05 to 0.2 percent of an organic dithiocarbamate selected from the group consisting of zinc, cadmium and iron dithiocarbamates.

2. The turbine oil of claim 1 in which said dithiocarbamate is zinc di-n-butyl dithiocarbamate.

3. The turbine oil of claim 1 in which said dithiocarbamate is cadmium di-n-butyl dithiocarbamate.

4. The turbine oil of claim 1 in which said dithiocarbamate is ferric di-n-butyl dithiocarbamate.

5. A lubricating composition comprising a lubricant base selected from the group consisting of mineral lubricating oil and a dicarboxylic acid diester lubricating oil and a minor proportion, sufficient to impart extreme-pressure properties to the lubricant, of a compound capable of releasing reactive sulphur selected from the group consisting of organic disulphides and polysulphides, together with from 0.01 to 1.0 percent of an organic compound selected from the group consisting of 2-mercaptobenzothiazole, benzothiazole disulphide, hydroxymethylthiobenzothiazole, and 2-mercapto 4,6,6-tri-methyl-1,3,6 H thiazine; and from 0.01 to 1.0 percent of an organic dithiocarbamate selected from the group consisting of zinc, cadmium and iron dithiocarbamates.

6. The lubricating composition of claim 5 in which said extreme-pressure imparting compound is an ester of acetic acid having the general formula

$$ROOC.CH_2—(S)_n—CH_2.COOR$$

in which $n$ is an integer from 2 to 4 inclusive.

7. The lubricating composition of claim 5 in which said derivative of said organic compound is benzothiazole disulphide.

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