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## METHOD OF PREPARING LUBRICANTS

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## ABSTRACT OF THE DISCLOSURE

This invention relates to a novel lubricant composition and a method for preparing the same wherein an inorganic salt solution is admixed with a solid lubricant.

The present invention relates to a method of preparing lubricants exhibiting a layer lattice structure and having improved adherence to the surface to be lubricated.

It is known that solid lubricants having a layer lattice structure can be incorporated in fluid or paste-like carriers or in lacquers. Among the solid lubricants so employed one might mention graphite; sulfides of metals of Group II of the periodic table of elements and particularly zincs and cadmium sulfides; inorganic fluorides such as calcium fluoride and sodium-aluminum fluorides such as cryolite. The lubricants containing the solid materials noted above suffer serious disadvantage in that these solids do not adhere to the surface to be lubricated, especially in dry form. Thus mechanical operations such as lapping, energetic rubbing and buffing and the addition of adhesion fostering materials are required.

It is apparent the lack of adequate adhesion of the lubricants to the surfaces results in serious loss of lubricity. Thus, corrosion appears when graphite is employed as a dry lubricant in the test method on the Almen-Wieland machine under a 50 kg. load.

It is the object of this invention to modify solid lubricants having a layer lattice structure to improve the adhesion of such lubricants to solid surfaces. A novel lubricant is also an object of this invention. Other objects and advantages of this invention are detailed in or will be apparent from the disclosure and claims following.

This invention relates to solid lubricants having a layer lattice structure, particularly those in dry form, treated with inorganic anions in the presence of an anion solvent.

The solid lubricants employed herein are those characterized by layer lattice structure. Examples of such solid lubricants include graphite, sulfides of metals from Group II of the periodic system of elements, particularly zinc sulfide and cadmium sulfide, inorganic fluorides such as calcium fluoride and cryolite (sodium-aluminum fluorides). Molybdenum disulfide and tungsten disulfide are not particularly useful in this invention, the results therewith being marginal.

The solid lubricants employed herein are treated with inorganic anions. The anions employed include the sulfide, sulfite, thiosulfate, sulfate, persulfate, halides and cyanammonium groups in particular but chromates, permanganates and other inorganic anions are also useful. Mixtures of anions can be used with excellent results. Thus a mixture of thiosulfate and fluoride gave exceptionally good results.

In contrast to the inorganic anions, the cations have little or no effect on this procedure. The solid lubricant and anion are brought into contact with each other in a solvent for the anion hence it is preferred to employ alkali metal, alkali earth metal or ammonium salts of the anions. These salts have been found to give excellent results in water solution and because of the economic ad-

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vantage inherent in employing water as the solvent, this system is preferred.

In addition to water, the solvents for the anions wherein the desired reaction is carried forward include organic as well as inorganic solvents having high dielectric constants. Operable solvents include aqueous ammonia, dioxane, alcohols, particularly lower aliphatic alcohols and mixtures of the foregoing as well as water solutions of such solvents.

The solid lubricant is contacted with the anion in the solvent and thereafter the solvent is removed by any desired means. The solvent can be removed by heat, vacuum, a combination of heat, a vacuum or even by physical means such as filtering and decanting in some instances.

The dry residue obtained will generally be in the form of a cake or gel and can readily be pulverized, powdered or comminuted by known means. Of course, the solvent may be retained and the lubricants can then be employed as dispersions or pastes. This is particularly useful when the solvent employed also acts as a carrier for other materials to be employed with the lubricant. However, when the solvent is retained it should not have a deleterious effect on the stability, lubricity and other characteristics of the lubricant.

The process of this invention permits the preparation of suitably compounded solid lubricants depending upon the purpose desired. In fact, the basic concept of the invention contemplates the use of solid lubricants of the layer lattice structure after treatment with inorganic anions in the presence of anion solvents. Thus the products of the present discovery can be used directly as dry lubricants alone or together with the common lubricant additives, in fluid or paste like carriers, lacquers, plastics, if desired in combination with other known lubricants and/or lubricant additives. The solid lubricants charged with anions in a quantity of from 0.2 to 4.0 percent by weight, preferably 1-2 percent, calculated on the dry weight of the solid lubricant have been especially useful for this.

The following examples are included herein to assist those skilled in the art to gain a full understanding of this invention. The scope of the invention is delineated in the claims and is not limited by the examples.

## EXAMPLE 1

Sodium thiosulfate was dissolved in water and powdered graphite was added to the solution. The resulting solution was tested as a lubricant on the Almen-Wieland machine and compared to other materials in this machine. The results are tabulated below:

| Lubricant                                                              | Load<br>(Kg.) | Torque (Final<br>Frictional<br>Force in Kg.) |
|------------------------------------------------------------------------|---------------|----------------------------------------------|
| A. Untreated graphite.....                                             | 50            | (1)                                          |
| B. Sodium thiosulfate powder.....                                      | 300           | (1)                                          |
| C. Dry graphite mixed with sodium thiosulfate powder in 1:1 ratio..... | 550           | (1)                                          |
| D. Graphite treated with sodium thiosulfate in 1:1 ratio in water..... | 2,000         | 300                                          |
| E. Same as D at 5:2 ratio.....                                         | 1,700         | 350                                          |
| F. Same as D at 2:3 ratio.....                                         | 1,750         | 350                                          |

<sup>1</sup> Break.

## EXAMPLE 2

Sodium sulfite was dissolved in water. Graphite powder was added to the solution to give a 1:1 ratio of sodium sulfite and graphite in the solution. The treated graphite and untreated graphite were tested in an Almen-Wieland machine wherein the untreated graphite broke under a 50 kg. loading while the treated graphite had a torque of 310 kg. at a 2000 kg. loading.

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## EXAMPLE 3

Ammonium sulfide was dissolved in water and powdered graphite was treated with the solution in a weight ratio of 1:1 of sulfide compound to graphite. The treated graphite was compared to untreated graphite in an Almen-Wieland machine. The treated graphite had a torque of 327 kg. (final frictional force) at 2000 kg. loading in contrast to the breakage of the untreated graphite at a load of 50 kg.

## EXAMPLE 4

Graphite powder was treated with cryolite in a weight ratio of 1:1 in a 30% soda lye solution. The Almen-Wieland test as in Example 1 showed the treated graphite was an excellent lubricant having a final frictional force of 350 kg. at a loading of 1950 kg.

## EXAMPLE 5

Sodium fluoride is dissolved in a suitable quantity of water and graphite powder is treated with this solution, wherein the weight ratio of sodium fluoride to graphite powder is 1:1. A final frictional force of 300 kg. is obtained with a load of 2,000 kg. on the Almen-Wieland machine.

## EXAMPLE 6

Calcium cyanamide is dissolved in water and graphite powder is treated with this solution, wherein the weight ratio of calcium cyanamide to graphite powder is 1:1. A final frictional force of more than 350 kg. at 1,200 kg. load is obtained on the Almen-Wieland machine.

## EXAMPLE 7

Potassium bichromate is dissolved in water and graphite powder is treated with this solution, wherein the weight ratio of potassium bichromate to graphite powder is 7:10. Although scoring of the shaft occurs at 50 kg. load on the Almen-Wieland machine with the graphite powder, the scoring occurs only at 1,000 kg. load when using the lubricant prepared according to the present discovery.

## EXAMPLE 8

Sodium fluoride is dissolved in water and cryolite powder is treated with this. The weight ratio of sodium fluoride:cryolite is 1:1. Wave breakage on the Almen-Wieland machine occurs at 1,000 kg. load while with cryolite the break occurs at 500 kg. load.

## EXAMPLE 9

Sodium fluoride is dissolved in water and zinc sulfide powder is treated therewith. The weight ratio of sodium fluoride:zinc sulfide is 1:1. Although shaft scoring occurs with zinc sulfide on the Almen-Wieland machine at only 250 kg. load, such scoring does not occur until a 750 kg. load with the lubricant prepared according to the present discovery.

## EXAMPLE 10

Sodium thiosulfate is dissolved in water and graphite powder is treated with this solution. The weight ratio of sodium thiosulfate to graphite is 1:1. Rings of the Alpha Molykote Lubricant Tester Model LFW-1 (see U.S. Patent No. 3,028,746) are coated with the completely dried product. At a total load of 290 kg. and a maximum drive coefficient set at 0.3 there were an average

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of 12,740 revolutions. The same graphite powder, not treated according to the discovery, yielded only 1470 revolutions under the same conditions.

## EXAMPLE 11

A paste is prepared from the solid lubricant of Example 1D by thoroughly mixing it with the same white oil. A load of 2,000 kg. on the Almen-Wieland machine yields a final frictional force of 226 kg. while the same paste made from graphite not treated according to the present discovery does not prevent corrosion under these conditions.

## EXAMPLE 12

A 5% suspension is prepared from the solid lubricant prepared according to Example 1D in mineral oil Rubrex 200. A final frictional force of 298 kg. is obtained on the Almen-Wieland machine under a 2000 kg. load, while severe scoring of the shaft occurs with the use of a 5% suspension of graphite powder which was not treated according to the present discovery, at a load of 850 kg.

That which is claimed is:

1. A method for preparing lubricants comprising

(a) dissolving an inorganic salt of a cation selected from alkali metals, alkali earth metals and ammonium and an anion selected from the group consisting of sulfides, sulfites, sulfates, thiosulfates, cyanamides and fluorides in a solvent selected from water, aqueous ammonia, dioxane, lower aliphatic alcohols, and mixtures of the foregoing, and

(b) admixing the solution from (a) with a solid lubricant selected from the group consisting of graphite, zinc sulfide, cadmium sulfide, calcium fluoride and cryolite.

2. A lubricant composition which comprises in synergistic proportions (a) an inorganic salt of a cation selected from alkali metals, alkali earth metals and ammonium and an anion selected from the group consisting of sulfides, sulfates, sulfites, thiosulfates, cyanamides and fluorides in a solvent selected from water, aqueous ammonia, dioxane, lower aliphatic alcohols and mixtures thereof, and (b) a solid lubricant selected from the group consisting of graphite, zinc sulfide, cadmium sulfide, calcium fluorides and cryolite.

3. The lubricant composition of claim 2 further defined in that the solid lubricant contains 0.2 to 4 percent by weight of the anion.

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