

## UNITED STATES PATENT OFFICE

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## TIMEPIECE LUBRICANT

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This invention relates to lubricants and more particularly to compositions for lubricating bearings and pivots of fine watches, chronometers, and timepieces generally, as well as bearings of moving parts of other precision instruments which present substantially similar lubrication problems.

It has been the practice for many years to employ natural oils taken from the head case of the blackfish, blubber, and the jaw of the porpoise for the lubrication of watches and other timepieces. These natural oils, properly refined by secret processes known only to a few families in this country and abroad, were the only satisfactory timepiece lubricants until the advent of "Elgin" watch oil a few years ago. Both the natural oils and Elgin oil have been preferred over other substances because of their high resistance to oxidation and their consequent freedom from gumming, even after long periods of exposure to air. Neither of these types of oils, however, has the ability to flow freely at low temperatures and, in fact, they solidify at temperatures well above those which are encountered under many more or less normal conditions of usage. It is a fact, for example, that neither of these oils serves satisfactorily in aircraft usage because of their tendency to form solids at the temperatures encountered, and thus to lose their lubricating qualities.

It is a further characteristic of these oils that they tend to spread to a greater extent than is desirable. This means, of course, that they creep away from the bearings to adjoining surfaces where they are not wanted. Since watches and other timepieces are rarely lubricated more frequently than once a year it is obviously desirable that the lubricant shall stay where it is put, namely on the frictional surfaces of the bearings.

A further objection to the prior art watch oils is their tendency to corrode metal parts. In a watch or chronometer this factor can be of major importance for even minute corrosion can soon render a timepiece unserviceable. Finally, there is the matter of cost of these previously used lubricants. A mere statement of the source of the natural oils indicates their scarcity and their consequent high selling price. Porpoise jaw oil, for example, costs several hundred dollars per gallon, and the Elgin oil, whatever its source, is just about equally expensive.

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The principal object of the invention is to provide a lubricant having a viscosity which is consistent with the requirements of timepiece lubrication, and which has so low a pour point that it stays in stable liquid form at temperatures far below those encountered under the most extreme conditions of timepiece operation.

It is a further object to provide a timepiece lubricant which tends to stay where it is placed and spread or creep away only to a very minor degree. In this respect, the object is to provide a liquid lubricant having far greater immobility, that is to say, lesser spread than exists in any previously known lubricant for the same class of service.

Another object of the invention is to provide a lubricant which is blended from synthetically produced ingredients that are readily available so as to allow for production at a reasonable cost and to insure duplication of the properties of each batch which is prepared.

We have found that the foregoing and other objects are met by a lubricant consisting essentially of from 60 to 80 per cent of tricresyl phosphate, from 25 to 7 per cent of ethylene glycol monoethyl ether ricinoleate, and from 15 to 13 per cent of triethylene glycol di-2-ethylbutyrate, all of said percentages being by weight with respect to the entire composition. It is to be noted that the tricresyl phosphate ingredient is a commercial mixture of the meta and para isomers of this compound, which is sold by the Celanese Corporation under the trade name of "Lindol (M. P.)." In general, the mixture is held to a limit of from 60 to 70 per cent of the meta isomer, and from 40 to 30 per cent of the para form, has a specific gravity at 68° F. of  $1.172 \pm .005$ , and a pour point of approximately  $-13^{\circ}$  F. For the sake of convenience, this mixture will be referred to both in the specification and claims as "tricresyl phosphate (M. P.)."

In view of the nature of the work which the lubricant is to perform we prefer to add a corrosion inhibitor to insure that the composition will not attack metals to more than a very slight extent. In this connection we have found that about  $\frac{1}{4}$  of one per cent of dicycloamine soap of isoamyloctyl acid orthophosphate serves very satisfactorily. When this is added, the propor-

tions of the major ingredients mentioned above are reduced appropriately.

The resulting compositions are clear, stable liquids, the several ingredients remaining in solution over any ordinary range of temperatures. All of the solutions within the limits given above, have excellent lubricating properties, and viscosities at 100° F. and at 50° F. which are entirely suitable to timepiece lubrication. In addition, and as indicated above, the corrosion characteristics of the lubricant vary from none to very slight, and the liquids are so low in volatility that their evaporation rates may be considered negligible.

The tendency of the compositions to spread on metal is extremely low. It varies to some extent with the variation in percentages of tri cresyl phosphate (M. P.) and the ricinoleate, from a low of less than 2 per cent to approximately 95 per cent, this factor being determined in the usual way by placing a drop of liquid on a piece of metal and gauging its increase in area over a period of time at predetermined temperatures. An appreciation of the smallness of spread within the ranges mentioned above can be gained from a consideration of the spread of other similar oils. Thus, the spread factor for ordinary household oils is on the order of 1000 per cent, while any normal penetrating oil will spread approximately 10,000 per cent. Oxidation and hydrolysis will likewise vary with the proportions of the phosphate and ricinoleate from a low of about 8 per cent to a high of slightly more than 30 per cent. It is accordingly possible, by an adjustment of proportions of these two ingredients, to produce a lubricant having approximately the spread required to meet any particular problem and at the same time its gum-forming properties can be kept as low as desired.

#### Example I

One example of a lubricant embodying the invention has the following composition:

|                                                 | Spec. Gravity | Per cent |
|-------------------------------------------------|---------------|----------|
| Tricresyl phosphate (M. P.).....                | 1.17          | 78.30    |
| Ethylene glycolmonoethyl ether ricinoleate..... | .929          | 8.24     |
| Triethylene glycol di-2-butyrate.....           | .995          | 13.24    |
| Corrosion inhibitor.....                        |               | .22      |

Any suitable corrosion inhibitor may be employed for its normal function of reducing the corrosive effects of the lubricant on metal parts. In the particular composition described above, we employed the agent referred to hereinbefore. Other specific and well-known inhibitors such as thio beta naphthol, diphenol disulphide, triphenyl bismuth, or the like may, however, be used if desired.

This composition when subjected to various forms of tests in the manner required by the Na. Aer. Spec. No. M537 exhibited the following properties:

Test designation:

Kinematic viscosity:

At 100° F. ....centistokes... 23.35

At 50° F. ....do..... 105.53

Solidification point.....°F... -60

Pour point.....°F... -45

Spread on metal.....per cent... 1.58

Oxidation and hydrolysis

per cent—no sediment... 8.12

Corrosion ..... None

Evaporation loss.....gram per day... .0006

#### Example II

Another lubricant which has been prepared in accordance with the invention has the following composition:

|                                                 | Specific Gravity | Per cent |
|-------------------------------------------------|------------------|----------|
| Tricresyl phosphate (M. P.).....                | 1.17             | 59.75    |
| Ethylene glycolmonoethyl ether ricinoleate..... | .929             | 25.00    |
| Triethylene glycol di-2-butyrate.....           | .995             | 15.00    |
| Inhibitor.....                                  |                  | .25      |

Tests of this lubricant composition carried out in accordance with the Na. Aer. Spec. No. M537 showed these properties:

Kinematic viscosity:

At 100° F. ....centistokes... 22.67

At 50° F. ....do..... 101.89

Solidification point.....°F... -70

Pour point.....°F... -55

Spread on metal.....per cent... 95.0

Oxidation and hydrolysis

per cent—no sediment... 30.2

Corrosion ..... Slight

Evaporation loss.....gram per day... .0002

The first of the exemplar compositions is particularly useful for the lubrication of the pivots and jewel bearings of watches, chronometers, etc. This follows from the fact that the lubricant has an extremely low spread factor, thus tending to stay in the place where it is put for long periods of time. In addition, it is highly resistant to oxidation so that it does not form gums which would clog the mechanism, nor does it corrode metal even to the slightest degree. Another factor of importance is its low evaporation loss, which, coupled with low spread factor, means that the lubricant will remain effective for very long periods of time, allowing for increase in the time of the normal lubrication cycle.

The second specific composition has a much higher spread factor than the first, and is not therefore quite so desirable for pivot lubrication. This factor is, however, an extremely low one for a lubricant and the composition serves very well for lubricating the main springs of watches and chronometers where creepage within limits is a desirable quality rather than otherwise. This second composition is more advantageous than the first one in that its pour point is lower. Thus it remains a clear liquid at temperatures down to -55° F. This means, of course, that there is no separation of the several ingredients at temperatures above -55° F., so that the liquid retains all of its original lubricating characteristics. This low pour characteristic, along with the fact that the liquid does not solidify until a temperature of -70° F. is reached, gives full assurance that the spring will not stick or bind under any ordinary operating conditions.

Having described our invention in its preferred form, what we claim as new and useful is:

1. A lubricant for timepieces and the like comprising from 60-80 parts by weight of tricresyl phosphate (M. P.), from 25-7 parts by weight of ethylene glycolmonoethyl ether ricinoleate, and from 15-13 parts by weight of triethylene glycol di-2-ethylbutyrate.

2. A lubricant for timepieces and the like comprising from 60-80 parts by weight of tricresyl phosphate (M. P.), from 25-7 parts by weight of ethylene glycol monoethyl ether ricinoleate, from 15-13 parts by weight of triethylene glycol di-2-

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ethylbutyrate, and about 0.25% parts by weight of di-cyclo amine soap of iso amyl octyl acid orthophosphate to inhibit corrosion.

3. A liquid lubricant for timepieces consisting essentially of about 79 per cent by weight of tricresyl phosphate (M. P.), about 8 per cent by weight of ethylene glycol monoethyl ether ricinoleate, and about 13 per cent by weight of triethylene glycol di-2-ethylbutyrate.

4. A liquid lubricant for timepieces consisting essentially of about 60 per cent by weight of tricresyl phosphate (M. P.), about 25 per cent by weight of ethylene glycol monoethyl ether ricinoleate, and about 15 per cent by weight of triethylene glycol di-2-ethylbutyrate.

5. A liquid lubricant for timepieces according to claim 3 characterized by about 0.25% of di-cyclo amine soap of isoamyl octyl acid orthophosphate.

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6. A liquid lubricant for timepieces according to claim 4 characterized by about 0.25% by weight of a dicyclo amine soap of iso amyl octyl acid orthophosphate.

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