Patented July 1, 1941 2,247,577

UNITED STATES PATENT OFFICE

2,247,577

LIQUID GREASE AND LUBRICANT

Marcellus T. Flaxman, Wilmington, Calif., assignor to Union Oil Company of California, Los Angeles, Calif., a corporation of California

No Drawing. Application March 14, 1938, Serial No. 195,819

15 Claims. (Cl. 252—34)

This invention relates to lubricants and primarily to the so-called liquid greases or kindred lubricants. The object of the invention principally is to produce an initially freely fluid lubricant which may be introduced to parts to be lubricated, and will penetrate into remote bearing portions of mechanical parts and will thereafter become more viscous or of a semi-solid or grease-like consistency for the purpose of retention of the lubricant.

According to the present invention, the desired property of fluidity in otherwise liquid grease or other lubricants not otherwise possessing sufficient fluidity, is imparted through the medium of a constituent which initially renders the lubricant liquid but eventually volatilizes or otherwise loses its liquefying properties so as to leave the lubricant in a more viscous condition than when applied or in a grease-like or similar form. According to a preferred form of the present invention, the constituent added is morpholine, but other amines having equivalent or approximately equivalent properties also may be employed.

Briefly stated, the invention may be said to reside in a lubricant containing a soap, especially an aluminum fatty acid soap such as aluminum stearate, in quantity sufficient to impart ordinarily a liquid or solid grease characteristic, said lubricant containing a small percentage of morpholine, for example between about 0.1% and 3%, either as such or in combination with fatty acids, to impart fluidity to the lubricant so that it may penetrate or flow readily into remote regions to be lubricated wherein even at relatively low operating temperature in said regions the morpholine will volatilize to restore in the lubricated zones the original liquid grease or solid grease characteristic.

The invention also resides in the use of other amines having sufficient volatilizing characteristics at the temperatures where the lubricant may be employed, especially at higher than normal temperatures. In order to possess sufficiently high vapor pressures, these amines in general will have boiling points below about 150° C. and, aside from the cyclic secondary amine ether morpholine, will fall in the classes of alicyclic amines and aliphatic amines. The invention extends also to such amine-containing lubricants wherein blending agents are added of such character that the residual lubricant after volatilization of the amine will possess the stringy characteristic of the castor machine oil type; and the invention further extends to the indicated type of amine-containing lubricants which are thinned with relatively large proportions of diluent such as naphtha after the fashion of typical pentrating oils. This applies particularly to the preferred morpholine which greatly aids penetration of the type of oil-soap mixture here employed. The invention further resides in the use of mineral oil-soap mixtures containing amines, especially where they contain morpholine, where the oil is a highly refined light mineral oil adapted especially for the production of spray oil and spray oil emulsions for horticultural uses.

In practicing the present invention according to a preferential form thereof, a lubricating oil is thickened with a few percent, e. g. 3% to 6%, of aluminum stearate to produce a heavy bodied lubricant non-flowing at ordinary temperatures. Such a grease penetrates either with difficulty or not at all into lubricated zones of small capacity. By adding to such a grease a considerably smaller proportion of morpholine, for example 0.5% or in the order of 0.2% to 1% of morpholine, the lubricant becomes fluid at ordinary temperatures to such an extent that it will pour. The result is that this fluid, pourable, soap-containing, lubricating oil will penetrate into regions to be lubricated where grease will not penetrate. By reason of exposure to air or for other or kindred reasons, the lubricant loses its fluid consistency after an interval of time, due apparently to volatilizing of the morpholine.

Whether this phenomenon is due to combination with or neutralization of excess acidity which remains in the soap upon manufacture, or whether it is due to functioning of the morpholine in its original form as by mere solution of the morpholine in the oil (by direct solution or in part due to the mutual solvent action of the aluminum or other soap), or to both, or to some other cause, is not entirely clear and I do not wish to be bound by any theory. If the morpholine combines with excess acid, the resultant soap evidently decomposes as the product loses its fluidity. In any event the morpholine serves as a means to permit the deposit of the resulting grease in remote regions by fluidizing or liquefying the thickened lubricant whereas heretofore only an oil could be used. In other words, an oil thickened with soap in amounts sufficient to substantially increase its viscosity so as to produce an ordinary liquid grease or heavier grease could not heretofore be employed, but now may be employed by thinning or fluidizing with morpholine so that the product acts as a sort of penetrating oil.

It has also been established that if morpholine

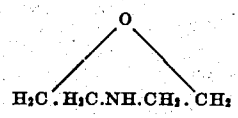
stearate be prepared and added to the above described aluminum stearate grease in approximately the same amounts as the morpholine is added, e. g. about 0.5%, the same or a very similar fluidizing effect is obtained, and that, if to this mixture a small amount of free morpholine be added, the phenomenon still exists.

In using the present type of composition, a more freely penetrating oil than that above indicated may be obtained either by reducing the quantity of soap, or increasing the quantity of morpholine, or both.

In addition to the above described liquid grease produced with aluminum stearate and morpholine or kindred amine, it may sometimes be desirable to produce a lubricant which will finally leave in the lubricating regions an oily material having the typical string of the so-called castor machine oils. In such a case, a blending agent may be used which in the absence of the morpholine or other amine would normally yield with the aluminum stearate and oil a stringy castor machine oil rather than a grease. Thus, when the fluidity which has been imparted by the morpholine has dissipated, a castor machine oil is left in the regions to be lubricated. For this purpose, an oil containing, for example, 4% or 5% of aluminum stearate and about 0.5% of morpholine to render the mixture fluid, will have added thereto about 1.5% (or between about 1% and 3%) of mono-butyl ether of di-ethylene glycol (known on the market as "butyl carbitol"), whereby when the fluid characteristics imparted by the morpholine have been lost a castor machine oil with the usual stringy characteristic will result. These stringy oils thus have penetrating properties imparted by the introduction of small quantities of morpholine as above indicated, and when the morpholine is volatilized, or the fluidity otherwise lost, the normal stringy castor machine oil type of lubricant remains.

Whatever the use to which any given composition is to be put, the object is to employ that quantity of soap and morpholine which represents the optimum amounts to secure maximum fluidity for the respective use. In any case, after the composition has penetrated to the regions to be lubricated, and a time has elapsed for the loss of fluidity by volatilization of the morpholine, or otherwise, as above indicated, a residual grease or similar deposit remains in the regions to be lubricated.

With respect to the use of morpholine for fluidizing the greases or liquid greases hereof, morpholine is the most desirable agent which I have been able to discover. It has the particularly desirable property of volatilizing out of solutions upon exposure to air or of otherwise losing its liquefying characteristics following deposit in the regions to be lubricated. This material has the empirical formula, $HN(CH_2CH_2)_2O$, and a structural formula



$H_2C.H_2C.NH.CH_2.CH_2$

As the formula shows, it has the characteristics of a cyclic ether and a secondary amine, and may be classed as a cyclic secondary amine ether. It has a boiling point of about 128° C., and its vapor pressure is so high that it directly volatilizes from the lubricant even at ordinary temperatures. For these reasons it can be employed in the preparation of penetrating lubricants of this type without regard to whether they are to be used at high temperatures or normal temperatures.

As a specific example of a fluid grease or lubricant produced according to this invention, 5% of aluminum stearate was dispersed in a lubricating mineral oil having a viscosity of 200 seconds Saybolt Universal at 100° F. The product was heavy bodied and non-flowing at ordinary temperatures. Into this normally solid grease, 0.5% of morpholine was incorporated, with the result that the product became sufficiently fluid to pour readily at ordinary temperatures. Upon exposure of this morpholine-treated grease to the air, thickening of the grease developed as the morpholine volatilized, and its original solid condition was reached after a reasonable lapse of time. Increasing the morpholine content to about 1.0% rendered the grease considerably more freely fluid, but increased the thickening period somewhat. Upon adding 1.5% of butyl "carbitol" to the lubricant containing 0.5% of morpholine, the material resulting after volatilization of the morpholine was a typical stringy castor machine oil.

This invention may also be applied to the preparation of so-called penetrating oils, wherein the initial viscosity of the lubricating oil is lowered by the addition of naphtha or the like; for example an ordinary mineral lubricating oil may be thinned with an approximately equal quantity of naphtha, and to this aluminum stearate and morpholine will be added after the fashion of the lubricants above described. Thus a suitable penetrating oil may be prepared from 50% naphtha, 47.5% of an average mineral lubricating oil such as one within the range of S. A. E. 10 to S. A. E. 60, 2% aluminum stearate and 0.5% morpholine. Such a penetrating oil, therefore, has its viscosity reduced to a markedly greater extent than when no morpholine had been added. Also, under ordinary circumstances, dilution of the oil-soap mixture with the naphtha would result in some of the soap being thrown out of solution to give an unstable product. The presence of the morpholine stabilizes the mixture which will remain clear and free from undispersed soap.

In the preparation of liquid greases of the types herein described, no full equivalent for the morpholine has been found, at least for ordinary uses, as above indicated. However, it may be possible to employ certain other amines as substitutes in special connections. For example, where these liquid greases are to be employed at higher than normal temperatures, it may be possible to use, instead of the cyclic secondary amine ether morpholine, the classes of amines consisting of other cyclic amine ethers, the alicyclic primary and other amines and the aliphatic primary and other amines, having boiling points below about 150° C. and sufficiently volatile. Of these materials I have found cyclo hexyl amine (boiling point about 134° C.) to be good, though not so good as morpholine. The mono, di and tri amyl amines are fair. Cyclic amines of the type represented by aniline, which has a very limited though definite value at least for some purposes, are the poorest group of usable amines. With respect to these amines it is to be noted that the tertiary amyl amine has a boiling point of about 78.5° C.; normal amyl amine has a boiling point of 104° C.; the secondary amyl amine has a boiling point of about 90° C. to 92° C.; iso amyl amine has a boiling point of about 95° C. and the secondary amyl amine has a boiling point of about 84° C. The hexyl amines, both normal and iso, have boiling

points of about 126° C. The boiling point of the above mentioned aniline is about 185° C.

Inasmuch as these materials cover the range of materials, other than morpholine, which may be used for some purposes, it is apparent that in general aliphatic amines and alicyclic amines having boiling points below about 150° C. are useful, especially such primary amines. Where the boiling points are higher, the degree of volatilization at normal temperatures, or in fact at most other operating temperatures, is too small. Even with respect to these indicated usable classes, the degree of volatilization from solutions is too small to offer the great value possessed by morpholine.

With respect to soaps which may be employed as substitutes for the aluminum stearate, it is ordinarily satisfactory to use other aluminum fatty acid soaps, or in fact any aluminum soap possessing sufficient solubility or compatibility in the mineral lubricating oil with which the grease or other lubricant is prepared. Examples of such other soaps are aluminum oleate, aluminum soaps of the coconut fatty acids, and in general the aluminum soaps of all those fatty acids containing from about 16 to about 20 carbon atoms per molecule. Also, the soaps of other metals may sometimes be employed, such as calcium oleate, where the soap in itself has a reasonable degree of solubility or dispensability in the oil to be used.

In addition to the use of morpholine or other indicated amine in the preparation of lubricants, it is contemplated that such amine may also be employed with the indicated types of soaps in the preparation of spray oils, wherein low viscosity well-refined petroleum oils, non-toxic to plant life, are prepared for use in water emulsions. For example, such a light mineral oil or spray oil containing from 1% to 5% of aluminum stearate as an emulsifying agent may have combined therewith from 0.2% to 1% (or on an average of about 0.5%) of morpholine or equivalent quantity of equivalent amine. Such a spray oil may be emulsified by agitation, with or without other constituents as well known in the spraying art, with about 98% of water to produce a 2% emulsion. When this emulsion is sprayed upon fruit or foliage, it breaks rather readily for the purpose of thorough distribution of the spray oil over the fruit and plant. When the morpholine volatilizes, the oil will thicken due to the presence of the aluminum soap and the oil will therefore tend to remain on the surface of the leaves and bark rather than to penetrate. Thus, it will remain where it can exert a maximum effect in insect and pest control.

Thus, the invention includes lubricants and the like containing metal soap, such as aluminum fatty acid soap, the soap being present in the order of 1% to 10% or even as high as 15%, and also containing an amine of indicated type in the order of 0.1% to 1% or even as high as 2%, which amine has a vapor pressure high enough to volatilize in places of use and preferably relatively high at normal temperatures, such as that of morpholine, and preferably having a boiling point below about 150° C. Also, soaps of the various amines, such as their stearic or other fatty acid soaps, may be used as substitutes for all or part of the amines per se, as above indicated in connection with morpholine stearate.

It is to be understood that the disclosures herein made are for the purpose of illustration of the invention and are not to be taken as limiting.

I claim:

1. Mineral lubricating oil containing between about 1% and 10% of an oil-soluble aluminum soap, and between about 0.5% and 3% of an organic normally liquid amine having high vapor pressure at normal temperatures and having fluidizing and spreading properties for the oil-soap mixture, the vapor pressure of the amine being high enough to volatilize from the lubricant in places of use at normal temperatures, the proportion of amine being sufficient to impart fluidity to the lubricant.

2. A fluid lubricant comprising mineral lubricating oil, soap to thicken the oil, and an amine normally liquid, having high vapor pressure and boiling below about 150° C. to render the oil-soap mixture freely liquid and volatilizable from the oil-soap mixture to increase the viscosity, the vapor pressure of the amine being high enough to volatilize from the lubricant in places of use at normal temperatures, the proportion of amine being sufficient to impart fluidity to the lubricant.

3. A lubricant comprising mineral lubricating oil, an aluminum soap to thicken the oil and a small proportion of morpholine to render the oil-soap mixture liquid.

4. A mineral lubricating oil containing between about 1% and 10% of an aluminum fatty acid soap adapted normally to impart grease-like characteristics and between about 0.1% and 3% morpholine to render the mixture liquid.

5. A mineral lubricating oil containing between about 1% and 10% of a metal soap to thicken the oil, and between about 0.1% and about 3% of an organic normally liquid amine of high vapor pressure having a boiling point below about 150° C. to render the oil-soap mixture liquid, the vapor pressure of the amine being high enough to volatilize from the lubricant in places of use at normal temperatures, the proportion of amine being sufficient to impart fluidity to the lubricant.

6. A fluid lubricant comprising mineral lubricating oil, a small proportion of soap in quantity sufficient normally to thicken the oil, and a small quantity of morpholine sufficient to render the oil-soap mixture liquid.

7. A mineral lubricating oil containing a quantity of aluminum soap sufficient normally to thicken the oil, and a small quantity of morpholine soap to impart fluidity to the oil-soap mixture.

8. A lubricant comprising mineral lubricating oil, a small quantity of fatty acid soap sufficient normally to thicken the oil, a small quantity of morpholine fatty acid soap and a small quantity of morpholine, the morpholine and the morpholine soap being in quantity sufficient to render fluid the otherwise thickened oil.

9. A lubricant comprising mineral lubricating oil containing between about 1% and 10% of aluminum fatty acid soap normally to impart grease-like characteristics, and a small quantity of morpholine fatty acid soap less than about 5% of the composition to render the composition liquid.

10. A lubricant comprising mineral lubricating oil containing between 1% and 10% of aluminum fatty acid soap to impart greasing consistency to the lubricant, a small quantity of morpholine fatty acid soap and a small quantity of morpholine, the combined morpholine and morpholine soap being less than about 5% of the

total composition and sufficient to render the mixture readily fluid.

11. Mineral lubricating oil containing between about 1% and 10% of a metal soap of a fatty acid containing at least about 10 carbon atoms and in quantity sufficient normally to thicken the oil, and between about 0.5% and about 3% of an organic normally liquid amine from the class consisting of cyclic amine ethers and alicyclic amines adapted to render the oil-soap mixture liquid and having relatively high vapor pressure at normal temperatures, the vapor pressure of the amine being high enough to volatilize from the lubricant in places of use at normal temperatures, the proportion of amine being sufficient to impart fluidity to the lubricant.

12. A lubricant according to claim 11 wherein the soap is aluminum soap and the amine has a boiling point below about 150° C.

13. A lubricant according to claim 11 wherein the amine has a boiling point below about 150° C.

14. A lubricant according to claim 11 wherein the soap is a calcium soap.

15. A lubricant comprising mineral lubricating oil containing in the order of about 1% to 15% of metal soap to impart a greasing consistency, and a small proportion in the order of 0.1% to 2% of an amine soap of an organic acid, the amine soap being adapted to render the oil-soap mixture liquid and the amine being normally liquid and having a relatively high vapor pressure high enough for volatilization of the amine from the lubricant in places of use of the lubricant and boiling below about 150° C., the proportion of amine soap being sufficient to impart fluidity to the lubricant.

MARCELLUS T. FLAXMAN.