

## UNITED STATES PATENT OFFICE

2,295,189

## SOAP AND LUBRICANT CONTAINING THE SAME

Reuben A. Swenson, Hammond, Ind., assignor to  
Standard Oil Company, Chicago, Ill., a corporation of Indiana

No Drawing. Application March 29, 1940,  
Serial No. 326,785

21 Claims. (Cl. 252—36)

This invention relates to a new soap, particularly adapted for use in lubricants, such as greases. More particularly it relates to greases containing this new composition of matter. Still more particularly it relates to the making of the new soap and to its use in making greases.

Soda soaps and lead-fish oil soaps and mixtures of the two are well known as bases for the manufacture of lubricants. For many purposes such lubricants have been found to be entirely satisfactory. However, under conditions of extreme pressure, particularly where high temperature is also encountered neither these lubricants nor those containing other bases have been found to be entirely satisfactory in use either from the point of view of lubrication or consumption. Moreover, lubricants which have otherwise satisfactory properties frequently do not have sufficient resistance to water or cannot be readily applied at normal temperatures.

It is an object of this invention to provide a new soap. Another object is to provide a method for making the new soap. A further object is to provide a new soap particularly adapted for use in lubricants. A still further object is to provide lubricants containing a new soap. A still further object is to provide lubricants having increased resistance to heat and water and increased adhesion adapting them for use under severe conditions of pressure and which may be readily applied at moderate temperatures. Other objects will appear hereinafter.

It has now been found that these objects may be accomplished by reacting sodium hydroxide with a soap formed by combining a heavy metal oxide and a fatty oil containing fatty acids having eight or more carbon atoms, preferably twelve or more, to produce a complex soap composition in which sodium, the heavy metal oxide, and the fatty oil are chemically combined. The preferred heavy metal oxide is litharge and particularly preferred fatty oils are mixtures of several fatty oils, especially mixtures including unsaturated fatty oils such as fish oils, for example, menhaden oil.

In the preparation of my complex soap I prefer to first make a heavy metal soap by reacting a heavy metal oxide such as lead oxide (litharge) with a fatty oil. This soap is made by reacting 3 moles of lead oxide or litharge with 1 mole of a fatty oil such as a fish oil. In order to prepare my complex soap hereinafter described it is desirable to have an excess of fatty oil present over that required to react 3 moles of litharge with 1 mole of the fatty oil. Since the soap becomes too

stiff to permit effective agitation during the reacting period it is desirable to add a diluent to the mixture in order to make the product more fluid. Where the product is to be compounded to make a lubricant a mineral oil of the desired viscosity may be used as the diluent. The viscosity of the mineral oil and the quantity used may be varied considerably depending upon the type of lubricant to be prepared. Preferably about 50% by weight of oil may be used but higher proportions as well as lower proportions ranging down to about 10% and lower may be used if desired.

A mixture of the fatty oil and the desired quantity of mineral oil is heated to about 300° F. and a small part of the required litharge is added with efficient stirring. As the reaction proceeds the litharge is added more rapidly while maintaining the reaction temperature at about 300° F. The mixture is agitated and maintained at this temperature until the reaction is completed. Lower temperatures may be employed but the time for completing the reaction will be materially increased. For the best results the temperature should not be permitted to exceed about 325° F.

The appropriate amount of lead oxide which should be employed with any suitable fatty oil can readily be calculated from the saponification number of the oil. The method of preparing the heavy metal component of my complex soap is described in detail in U. S. Patent No. 1,830,984.

Lead soap prepared in the manner just described is then converted into the new complex sodium-lead soap by reacting sodium hydroxide with the lead soap just described. This is accomplished by adding an excessive amount of sodium hydroxide over that required to saponify the additional fatty oil that is added in the preparation of the complex soap. Preferably the amount of caustic employed should equal about 1 mole of caustic per mole of heavy metal. The reaction between the sodium hydroxide and the lead soap takes place at an elevated temperature. The reaction may be initiated at temperatures of the order of about 150° F. to about 200° F. and thereafter the reaction is carried to completion at a temperature of about 400° F. By completing the reaction at the high temperature the reaction time is considerably reduced and any water present in the final product is substantially eliminated. In carrying out this reaction it is preferable to have a diluent present in order to facilitate efficient agitation of the reaction mixture. Such a diluent may be, for example, the petroleum oil called for by the lubricant formula

where the sodium-lead soap is being made for use in a particular lubricant.

While I have described the preparation of the complex sodium-lead soap the same has been used only by way of example, it being understood, of course, that in place of sodium hydroxide any one or more of the alkali or alkaline earth metal bases of groups I and II of the periodic table, such as calcium hydroxide, barium hydroxide or potassium hydroxide may be so employed.

Although the formation of the new complex sodium heavy-metal soap has been described above without regard to the preparation of any particular lubricant, in actual practice, as has been indicated, the new soap is usually formed in the course of the preparation of the lubricant in which it is intended to be used. The heavy metal oxide-fatty oil reaction product is, however, in the usual procedure made up in advance. It is desirable that the concentration of the heavy metal soap in the base oil be substantially at its maximum as it is easier to decrease this concentration in making up lubricants than it is to increase it. The lead fish oil soap, when so made up is very stiff and difficult to handle, and, as explained above, for uniform reaction and ease in handling, a diluent is desirable. Since petroleum oils having Saybolt Universal viscosities at 100° F. ranging from about 80 to about 2500 seconds are constituents of a large number of lubricants containing my complex alkali-lead oxide soap and also are well adapted for use as a diluent, the reaction between lead oxide and the fatty oil is usually carried out in the presence of an oil within this viscosity range. The quantity of such petroleum oil which should be used will depend upon a number of factors such as the viscosity of the oil, the formula of the desired lubricant, etc. A particularly convenient procedure where the soap is formed from litharge and menhaden oil using the maximum quantity of litharge, (i. e. about 80% by weight, based on the menhaden oil) is to have present as a diluent an amount of the desired petroleum oil equal in weight to the combined weight of the litharge and menhaden oil.

This base, identified as the lead-fish oil soap base, is then added to a substantial portion of the additional petroleum oil called for by the formula of the lubricant, preferably about an equal weight, in a vessel adapted to be heated, for example, a fire kettle, and heated preferably to about 400° F. or somewhat higher. The petroleum oil to which the base is added, may be, for example, a blend of about 50% to about 70% residuum (furol viscosity at 210° F. of about 800-900 seconds), and about 50% to about 30% low viscosity petroleum oil having a Saybolt viscosity at 100° F. of about 80-300 seconds. If additional fish oil or other fatty oil is to be used it is also added at this point.

The mixture is then heated, for example, to a temperature of about 160° to about 200° F. and sodium hydroxide is added. The quantity of sodium hydroxide should be substantially in excess of that required to saponify the excess fatty acid present in the 50% base. Preferably, as pointed out above, the excess of sodium hydroxide should be equal to 1 mole of sodium hydroxide for each mole of lead oxide present in the base. The excess sodium hydroxide may, however, if desired exceed even this amount to some extent.

Reaction takes place at the elevated temperature and is preferably accelerated by raising the

temperature to about 200° to 220° F. after the addition of sodium hydroxide. Still further elevation of the temperature, particularly after a heavy base is formed is also desirable. Thus, for example, after the heavy base is formed it is preferred that the temperature be raised gradually to about 400° F. or somewhat higher.

If the formula of the lubricant calls for ingredients not yet included they may be added by grading them in at this point. For example, the formula may call for additional petroleum oil over that already incorporated. During such additions the temperature is preferably kept high, for example, at about 400° F. In order that the mixing of the added materials may be completed it is desirable to maintain the elevated temperature and stir for about ½ hour or longer after the addition is complete.

The lubricant may now be filled at the high temperature to which it has been heated, for example, about 400° F. or at a lower temperature. A smoother product is usually obtained if the mass is stirred to about 300° F. to 250° F. before filling. Additional desirable characteristics such as stringiness may be obtained by adding high molecular weight synthetic polymers such as high molecular weight iso-olefin or mono-olefin polymers before filling. Fillers, such as asbestos, talc or graphite, may also be added before filling.

In order to illustrate exact methods for carrying out the invention the following examples are included. It will be understood that these examples are intended to be construed as illustrative only and not as limiting the scope of the invention.

#### EXAMPLE I

A 50% lead-fish oil soap base is first prepared by adding 12 parts by weight of litharge to 5 parts by weight of a light petroleum oil (Saybolt viscosity 95 to 100 seconds at 100° F., flash about 300° F.) in a steam jacketed positively scraped (single or double action) mixer and stirring until a smooth paste results. Twenty-two additional parts of the light petroleum oil and 15 parts of menhaden oil are then added rapidly, the steam is turned on and the contents of the mixer are heated at 300-325° F. for 4 hours, when the reaction is complete. The product is then filled into barrels for storage or it can be treated further immediately.

To make the complex sodium-lead-fish oil soap 31.8 parts of the 50% lead-fish oil soap, prepared as above, is added to approximately an equal weight of a suitable blending oil in a fire kettle. The contents of the kettle are heated to 160°-200° F. and 3 parts (dry weight) of sodium hydroxide solution of about 48° Bé. gravity are added. Reaction is continued at 200-220° F. until a heavy base is formed and then the temperature is gradually raised to 400° F. The resultant product is a highly heat resistant material which may be blended with other ingredients to produce lubricants suited for a wide variety of uses.

#### EXAMPLE II

A lubricant may be prepared by mixing in a fire kettle 31.8 parts of a 50% lead-fish oil soap base (prepared as described in Example I in a diluent comprising a low cold test stock having a flash of about 300° F. and a Saybolt viscosity at 100° F. of about 80-85 seconds) with approximately an equal amount of a mixture of 65 parts of a residuum stock having a Furol viscosity at

210° F. of about 850 seconds and 35 parts of a low cold test oil having a flash of about 300–315° F. and a Saybolt viscosity at 100° F. of about 80–85 seconds. Five parts of menhaden oil are then added and the contents of the kettle are heated to 150–200° F. Three parts (dry weight) of sodium hydroxide solution of about 48° Bé. gravity are then added and the temperature maintained at 200–220° F. until a heavy base is formed. Thereafter the temperature is raised gradually to approximately 400° F. to complete the reaction. The balance of the oil called for by the following formula is then graded in at about 400° F.

#### Formula

|                                                                                                                                      | Parts |
|--------------------------------------------------------------------------------------------------------------------------------------|-------|
| Lead-fish oil soap (50%)                                                                                                             | 31.8  |
| Menhaden oil                                                                                                                         | 5.0   |
| Sodium hydroxide                                                                                                                     | 3.0   |
| Winkler C. C. stock (residuum stock having a Furol viscosity at 210° F. of about 850 seconds)                                        | 49.5  |
| 300 flash L. C. T. stock (low cold test oil having a flash of about 300–315° F. and a Saybolt viscosity at 100° F. of 80–85 seconds) | 10.7  |

After all the oil has been added the batch is stirred as it cools to 300° F., at which temperature it is filled. The product is an excellent lubricant for heavy, loose running machinery, particularly gears exposed to high temperatures, such as the table roll gears, side roll gears, driving pinion gears and bevel gears, etc. found on steel mill machinery. Lubricants of this type are known in the art as gear shield lubricants. When heated to 500–600° F. on a hot plate it maintains its grease structure without flowing appreciably over an extended heating period almost to the point of carbonization. No similar type of lubricant is known which will maintain its structure at such a high temperature for even half the time that the product of this example will. Substantial neutrality and freedom from water also characterize the material. It is also non-fibrous, smooth and unctuous and adheres tenaciously to metal surfaces.

#### EXAMPLE III

The lubricant of Example II is quite satisfactory for use in moderate or warm weather but because of its high oil viscosity it is difficult to apply in cold weather. A better lubricant from the standpoint of cold weather application has the following formula:

|                          | Parts |
|--------------------------|-------|
| Litharge                 | 5.95  |
| Menhaden oil             | 11.8  |
| Sodium hydroxide (dry)   | 2.55  |
| 300 flash L. C. T. stock | 29.4  |
| Winkler C. C. stock      | 35.3  |
| Heavy black oil          | 15.0  |

The lubricant is formed in the same manner as that described in Example II by first making a 50% lead-fish oil soap with 5.95 parts litharge, 7.55 parts menhaden oil and 13.5 parts 300 flash L. C. T. stock. The 27 parts of lead-fish oil soap base are then combined with the other ingredients as in Example II.

The resulting product is of the same general nature as that produced in Example II except that it is softer and more free flowing at low temperatures. It has been found to be an ex-

cellent lubricant for leaf springs, particularly those equipped with pressure gun fittings. This was established in a test very closely simulating actual service conditions.

- 5 In conducting this test a spring is dismantled, cleaned with a petroleum solvent, dried and re-assembled. It is then placed in the testing machine which flexes the spring repeatedly. The spring is then lubricated with the grease under  
10 test and the machine is then started and run continuously until squeaking occurs. The results of the test were as follows:

| Lubricant*             | No. of hours before squeaking      |
|------------------------|------------------------------------|
| 15 A                   | 140.                               |
| B                      | 84.                                |
| C                      | 72.                                |
| Product of Example III | More than 214 (test discontinued). |

- 20 \*Lubricant "A" is a standard pressure lubricant now in wide commercial use.  
Lubricant "B" is made according to the formula of a large automobile manufacturer.  
Lubricant "C" is a special lubricant made up for test purposes.

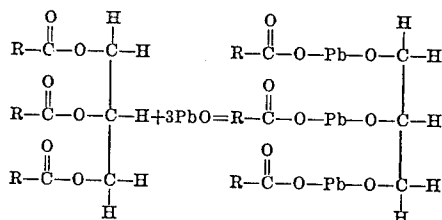
- 25 At the start of each test there is an initial period of squeaking that lasts until the lubricant spreads throughout the leaves. In the above test, this initial squeaking period was very short where the lubricant of Example III was used, indicating that it spreads rapidly between the  
30 leaves. The test on this product was discontinued after 214 hours since there was no practical value in running it any longer but examination of the springs leaves after the test disclosed an ample coverage of lubricant so that undoubtedly the test would have continued for a much  
35 longer period before squeaking would have occurred. Thus the test indicates that the new grease is superior both in its ability to spread rapidly between the leaves and in its ability to  
40 adhere tenaciously to the leaves during service.

- The product of Example III is not only a good spring lubricant, but is also a very good heavy gear shield lubricant for use during cold weather. The product of Example II is quite satisfactory  
45 at moderate or elevated temperature but at low temperatures is somewhat difficult to apply due to its high oil viscosity. Under these conditions the product of Example III has been shown to be quite satisfactory both as a lubricant under  
50 severe conditions of pressure and temperature as well as in ease of application at low temperatures. The products of both Examples II and III have been tested as heavy gear shield lubricants in actual service conditions in a steel mill  
55 and have both been found to be entirely satisfactory from both effectiveness in lubrication and in consumption.

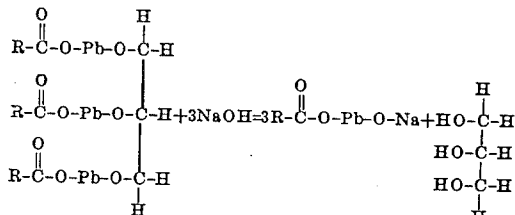
- The temperatures and times of reaction indicated above have been found to give satisfactory products, but these may be varied so long as they are adequate to bring about the formation of the new complex sodium-heavy metal soap.

- I am fully aware that mixtures of soda soaps and lead-fish oil soaps have heretofore been used in lubricants, particularly greases. My new product differs from the soaps heretofore used in that it is not a mixture of these soaps but is a complex soap. Without intending to limit the invention to any particular theory, it is believed  
70 the following reactions take place to form the complex soap of the present invention. The formation of the lead oxide-fatty oil soap is believed to take place according to the following formula, in which the heavy metal oxide is rep-

resented by lead oxide or litharge for the purpose of illustration



The lead soap—(lead-fish oil soap) reacts with the caustic according to the equation



That the above reaction probably takes place is borne out by the fact that substantially no lead glycerate appears in the final product. Furthermore lubricants prepared with neutral sodium-fish oil soap and lead-fish oil soap in admixture do not possess the heat resistant property of my improved product, or other desirable properties, although visually the products may appear to be the same.

In the lubricant formulae given above the variety of ingredients is not great but it will be apparent to those skilled in the art that many other materials may be included. For example, as indicated above, oil soluble high molecular weight polymers may be added to impart stringiness and fillers may also be incorporated in the lubricants and other lubricant materials such as graphite, may be present if desired. Petroleum oils of any desired characteristics and in proportions adapted to a large variety of service conditions are contemplated for use with the new soaps, particularly the soda-lead-fish oil soaps.

Many advantages of the new soaps and greases containing them are apparent from the foregoing. Thus it has been shown that the new soaps and greases containing them have excellent resistance to high temperature so that they are adapted for use at high temperature. Moreover, the lubricants containing the new soaps adhere tenaciously to surfaces where applied and are accordingly adapted for use under conditions of extreme pressure. Not only do they adhere tenaciously but when properly compounded they are easy to apply and spread rapidly between the surface to be lubricated even under conditions of extreme pressure. This particularly adapts these lubricants for use under conditions of extreme pressure where application is unusually difficult. Because of their tenacious adherence these lubricants are also especially suited for use on open loose running machinery where they are subject to a force such as centrifugal force tending to throw them away from the surfaces requiring lubrication. They are quite resistant to water and where this is a problem their use is further indicated.

It is apparent that many widely different embodiments of this invention may be made without departing from the spirit and scope thereof and, therefore, it is not intended to be limited except as indicated in the appended claims.

I claim:

1. The process which comprises reacting lead oxide and a fatty oil and subsequently reacting the product so obtained with an hydroxide of a metal selected from the class consisting of alkali metals and alkaline earth metals at a sufficiently elevated temperature to substantially eliminate any water present in the final product whereby a complex soap of lead and a metal selected from the class consisting of alkali metals and alkaline earth metals is formed.

2. The process of claim 1 further characterized in that the reactions are carried out in the presence of a hydrocarbon diluent.

3. A product obtained by the process of claim 1.

4. A product obtained by the process of claim 1 when the reactions are carried out in the presence of a hydrocarbon diluent.

5. A lubricant composition comprising a lubricating oil and the product obtained by the process of claim 1.

6. The process which comprises reacting lead oxide and a fatty oil containing long chain fatty acids in the presence of a hydrocarbon diluent and then reacting the product so obtained with an hydroxide of a metal selected from the group consisting of alkali metals and alkaline earth metals in the presence of a hydrocarbon diluent, said lead oxide being present in said first named reaction in an amount equal to from about 1.5 to about 3.0 molecular proportions to each molecular proportion of fatty oil.

7. A compound which is a reaction product of litharge, a fatty oil and sodium hydroxide obtained by the process of claim 6 and containing lead, the long chain of the fatty acid and sodium.

8. The product obtained by the process of claim 6.

9. A lubricant composition comprising a lubricating oil and the product obtained by the process of claim 6.

10. The process which comprises reacting litharge and a fatty oil and then reacting the product so obtained with sodium hydroxide at a sufficiently elevated temperature to substantially eliminate any water present in the final product whereby a complex sodium lead soap is formed.

11. A product obtained by the process of claim 10.

12. A lubricant composition comprising a lubricating oil and the product obtained by the process of claim 10.

13. The process which comprises reacting litharge and fish oil and then reacting the product so obtained with sodium hydroxide at a sufficiently elevated temperature to substantially eliminate any water present in the final product whereby a complex sodium lead fish oil soap is produced.

14. A product obtained by the process of claim 13.

15. The process which comprises reacting in the absence of water about three molecular proportions of lead oxide and at least one molecular proportion of a fatty oil containing fatty acids of at least eight carbon atoms to form a lead soap of said fatty oil and subsequently reacting the product so formed with a hydroxide of a metal selected from the class consisting of alkali metals and alkaline earth metals at a temperature of from about 220° F. to about 400° F.

16. The process as described in claim 15 in which the hydroxide employed is equivalent to one mole of hydroxide per mole of lead.

17. The process described in claim 15 in which the reaction is carried out in the presence of a petroleum oil as a diluent.

18. The product obtained by the process of claim 15.

19. A lubricating composition comprising a lubricating oil and the product obtained by the process of claim 15.

20. A lubricant composition obtained by reacting the following ingredients in the approximate specified proportions:

|                                                                                                                                      | Parts |
|--------------------------------------------------------------------------------------------------------------------------------------|-------|
| Lead-fish oil soap (50%)                                                                                                             | 31.8  |
| Menhaden oil                                                                                                                         | 5.0   |
| Sodium hydroxide                                                                                                                     | 3.0   |
| Winkler C. C. stock (residuum stock having a Furol viscosity at 210° F. of about 850 seconds)                                        | 49.5  |
| 300 Flash L. C. T. stock (low cold test oil having a flash of about 300-315° F. and a Saybolt viscosity at 100° F. of 80-85 seconds) | 10.7  |

in which the lead fish oil soap is prepared by reacting three moles of litharge with one mole of menhaden oil in the presence of a petroleum oil, said lubricant being prepared by adding a portion of the blended mineral oil together with the menhaden oil to said lead fish oil soap, heating the mixture to a temperature of about 150 to 200°

F., adding the sodium hydroxide to the mixture and heating the same to a temperature of about 200 to 220° F. until a heavy paste is formed, raising the temperature to about 400° F. and subsequently adding the remaining portion of said blended mineral oil.

21. A lubricant composition obtained by reacting the following ingredients in the approximate specified proportions:

|                           | Parts |
|---------------------------|-------|
| Litharge                  | 5.95  |
| Menhaden oil              | 11.8  |
| Sodium hydroxide (dry)    | 2.55  |
| 300 flash, L. C. T. stock | 29.4  |
| Winkler C. C. stock       | 35.3  |
| Heavy black oil           | 15.0  |

in which the litharge, 7.55 parts of the menhaden oil and 13.5 parts of the 300 flash L. C. T. stock are mixed at an elevated temperature until a smooth paste is formed, adding the remaining portion of the menhaden oil, a portion of the remaining mineral oil and the sodium hydroxide to said lead fish oil soap, heating the mixture to a temperature of about 200 to 220° F. until a heavy paste is formed, raising the temperature to about 400° F. to complete the reaction and subsequently blending in the remaining portion of the mineral oil.

REUBEN A. SWENSON.