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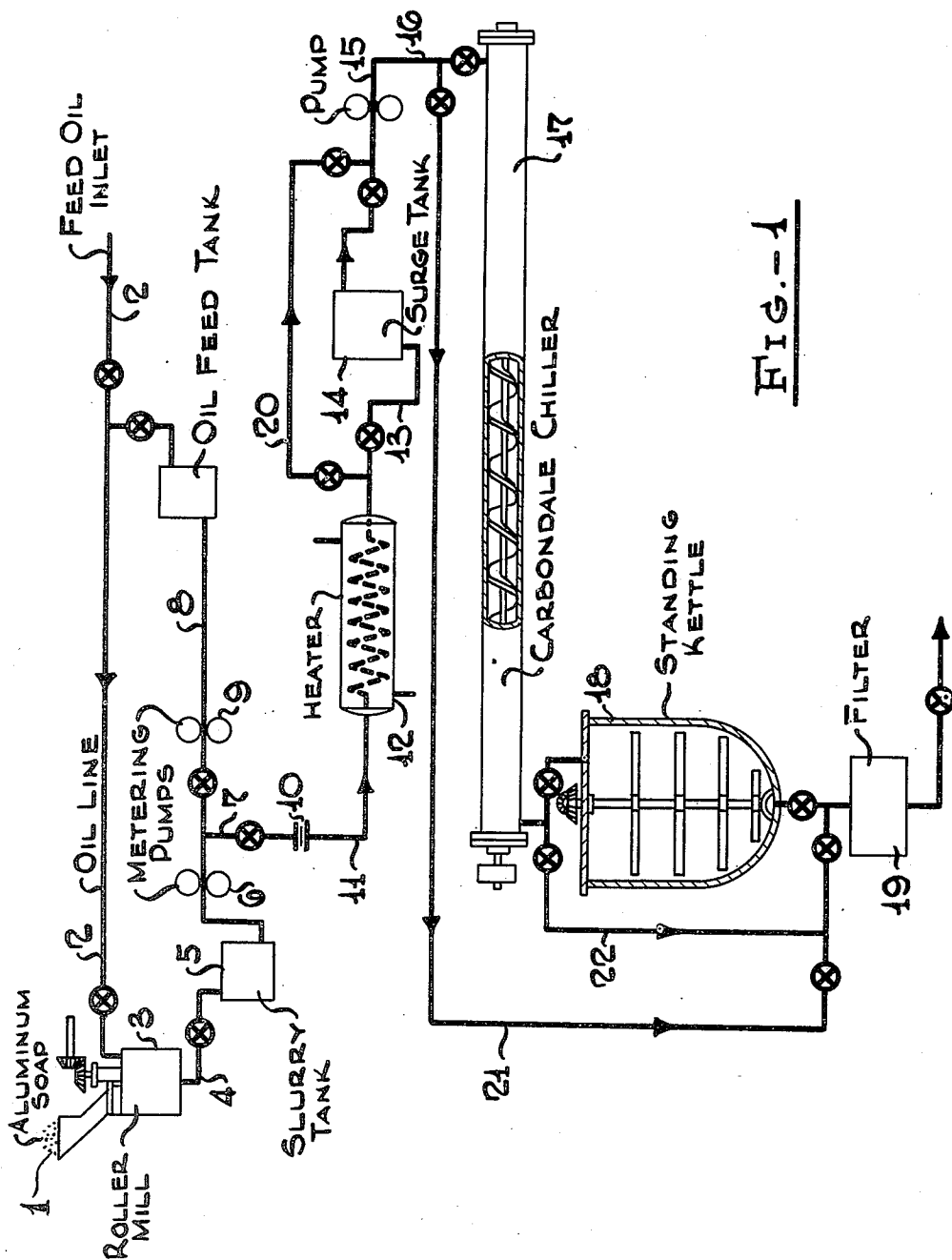
A. BEERBOWER ET AL

2,431,453

LUBRICANT MANUFACTURE

Filed Dec. 30, 1944

2 Sheets-Sheet 1



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2 Sheets-Sheet 2

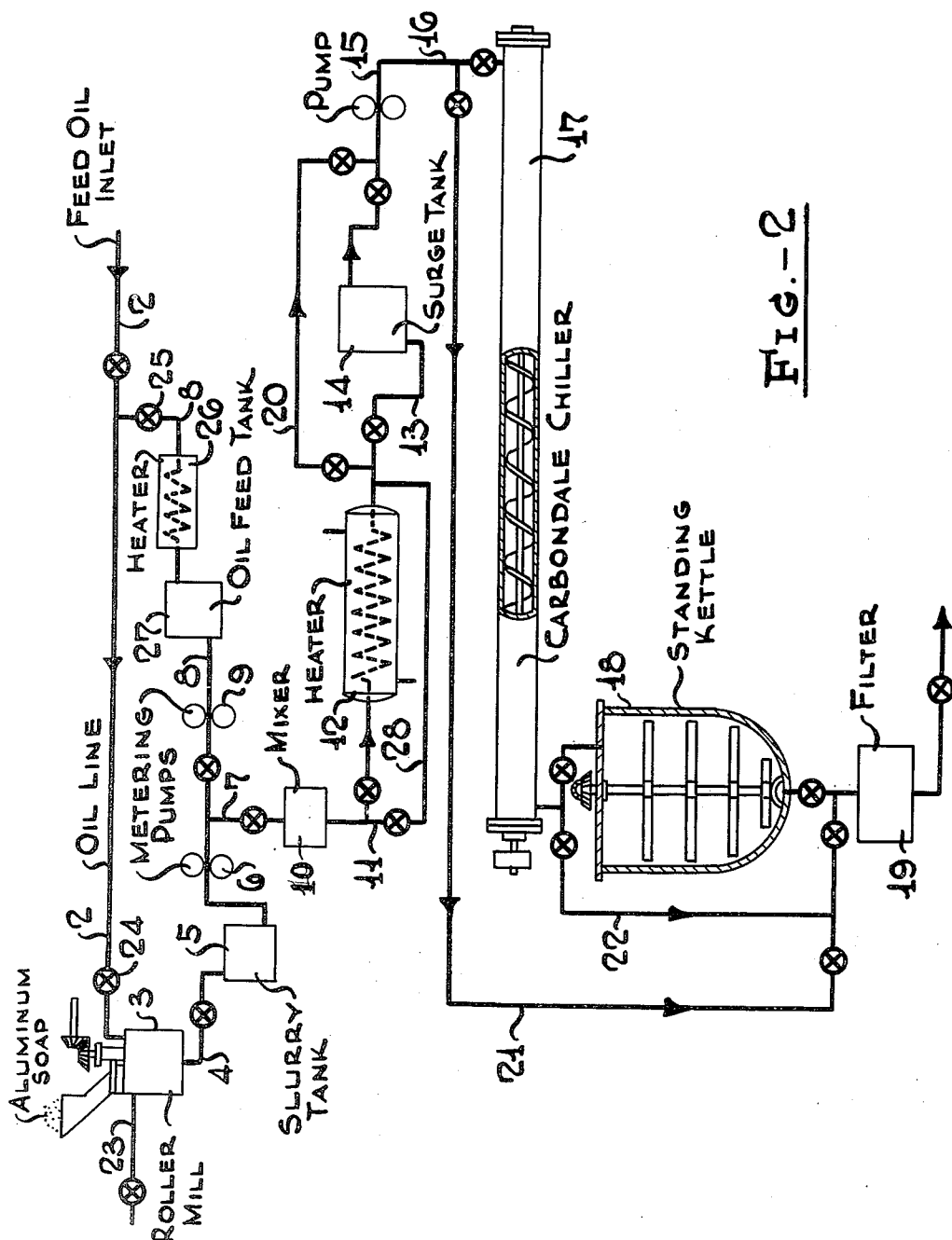


Fig. 2

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UNITED STATES PATENT OFFICE

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LUBRICANT MANUFACTURE

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8 Claims. (Cl. 252—35)

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This invention relates to an improved process and apparatus for manufacturing lubricating greases, especially aluminum soap greases. The invention is particularly adapted for continuous manufacture of such greases.

Many different attempts have been made to develop satisfactory continuous processes for manufacturing lubricating greases, but few of these methods have met with the desired degree of success, particularly due to difficulties which characterize the manufacture of certain types of greases, especially aluminum grease, and in certain respects soda, barium and lithium greases. One object of the present invention is to overcome these various difficulties of the prior art and to provide a process for the continuous manufacture of aluminum soap grease, as well as soda soap grease and even other soap greases, if desired, in simple and relatively fool-proof equipment which in operation results in a high quality homogeneous uniform and reproducible product at relatively low cost.

Broadly, the invention comprises mixing a soap and a lubricating oil at a temperature considerably below the maximum grease-cooking temperature to form a lump-free slurry, diluting said slurry with additional oil to give the proportions of soap and oil desired in the finished grease, mixing said materials to make a substantially homogeneous mixture, then passing the mixture through a heating zone of relatively large heating surface per volume, in order to get complete solution of the soap in oil with once-through operation, and cooling the resultant grease stock at least to a grease structure-forming temperature. The invention may be exemplified by mixing a dry aluminum stearate powder with oil in a sufficient amount to make a thick slurry, this mixing being preferably done in a roller or pug mill which breaks up lumps efficiently, although it may be done with an agitator in a barrel, then adding more oil, still without any substantial heating of the mass, mixing these materials thoroughly and passing them through a pipe coil heater to bring the mixture up to a suitable grease-cooking temperature, such as 250°–350° F. for an aluminum soap grease, or higher for other soaps such as soda or lithium or barium, then passing the materials to a surge tank from which accumulated vapors may be discharged, and finally cooling the mixture in a suitable manner to permit the formation of the desired grease structure. Usually in the past with aluminum soap grease this cooling has been generally effected by pouring the grease stock into a large number of relatively small

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shallow pans and allowing them to cool for a day or two, then returning to the kettle, working the product to a uniform consistency, and then packaging.

5 The preferred method is to pass the grease stock through a helical blade (or screw conveyor) chiller, preferably one cooling down to the transition temperature of the grease, and allowing it to remain there for the required transition period of a ¼ hour to six hours or so, or slowly
10 cooling the grease even on down past the transition temperature and allowing the grease to go through its final transition stages in a storage kettle or in shipping containers. This procedure
15 does not require floor space for storage and use of pans and it eliminates labor, shortens manufacturing time and produces greases of uniform and satisfactory consistency from lot to lot, since the cooling operation can be more closely controlled. Pan operation, where cooling is not controlled, frequently yields non-uniform products from pan to pan.

Instead of using aluminum stearate, other
25 aluminum soaps may be used such as aluminum naphthenate or aluminum soaps of other carboxylic acids having 10, or preferably 15, carbon atoms or more, such as aluminum soaps of synthetic fatty acids derived from the oxidation of paraffin wax, or mixtures of these various soaps,
30 such as a mixture containing about 90% to 95% of aluminum stearate and 10% to 5% of aluminum naphthenate. Naphthenic acid or some other suitable high molecular weight acid such as oleic acid, etc., alcohols, glycols, glycerine
35 esters, phenols, aliphatic or alkyl aryl amines or alkanolamines may be used alone or with aluminum naphthenate to modify the grease structure or change the rubbery liquid to solid gel transition temperature range attendant upon the
40 use of aluminum stearate.

The oil to be used both in making the original slurry and in subsequently diluting the slurry to the desired final proportion of oil in the finished grease may be any of the conventionally used
45 lubricating base stocks, such as various light, medium or heavy lubricating oil distillates or residual fractions which may have been subjected to any of the conventional refining treatments such as clay-treating, acid-treating, solvent extraction, etc., and may be derived from various crude oils
50 such as paraffinic, naphthenic, or mixed base crudes. In addition, other suitable fluid lubricants, fuels or liquids may be compounded with soap to yield pastes, solid fuels, etc.

55 The proportions in which the soap and oil

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should be mixed in order to make the slurry in the first step according to this invention, should be about 15% to 35% by weight of soap and 65% to 85% of oil, depending largely upon the viscosity and other characteristics of the oil used as well as the type of soap used.

Although the soap, aluminum, lithium, soda or other metal soap, which is preferably in the form of a dry powder, may be mixed with the oil in this first step without the use of any heat whatsoever, it is possible if desired to use oil which has been warmed slightly, e. g., up to 100° or 150° F., or so, to facilitate the mixing.

As suggested above, the slurry mixer should preferably be an efficient lump-breaking mixer, such as the usual roller mill, in which the periphery of a roller having a horizontal axis and a peripheral edge of substantial width, rolls in contact with the upper side of a disc roller rotating on a vertical axis, so that lumps in the material being mixed are crushed between the rollers. Other types of slurry mixers may be used such as a small grease kettle with rotating paddle blades, a bread-dough type of mixer or a parallel-roll paint mill. An effective type is also available in which two interlocking rotors knead the mass together.

Of the various types of slurry mixers described above, the Lancaster roller mill is preferred. This is described briefly as follows: The mixer tank rotates about one axis and the mixer blades about another eccentric to the first. The blades consist of both "plows" to lift the mass and "mullers" to force it against the tank bottom.

The mixer in which the slurry is subsequently mixed or diluted with more oil, which for the sake of clarity may be referred to as the blending mixer, is preferably one which is suitable for continuous operation, such as the orifice mixer. Other types of blending mixers may be used, however, such as gear or rotor pumps, in which controlled amounts of the slurry and oil are fed in together and rapidly mixed and discharged in a continuous operation, the amount of recycling of the material through the gear being controlled at will. A centrifugal pump or a rotary mixer of the colloid mill type or the Lancaster disperser type may also be used.

The heater through which the blend of slurry and oil is then passed should preferably be a pipe coil heater having sufficient inside pipe diameter to give the desired capacity, and of sufficient pipe length to give the required heating in a once-through operation. The surface of the pipe coil heater should be at least 4 square inches per cubic inch of volume and the temperature of the grease at the outlet should be between the approximate limits of 200° and 500° F., depending upon the type of grease being produced, for instance about 250° to 350° F. for aluminum, magnesium or barium soap, preferably about 280° to 300° F., and on the other hand about 350° to 500° F. for sodium or lithium soap grease, and about 200° to 250° F. for calcium, lead or zinc soap grease. Other types of heaters suitable for continuous operation are those containing a rotating scraper to insure good heat transfer, or a tube bank variety.

On the other hand, for batch operation an ordinary kettle without wall scrapers is suitable either for use with high pressure steam or heated by direct firing.

Although the surge tank mentioned above is not necessary in all cases, it is a desirable accessory in the equipment in order to provide for venting of accumulating gases and vapors, and

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also in order to even out any irregularities in the volume of flow of the materials in the various parts of the equipment before or after the surge tank.

If it is desired to cool the grease, this is preferably done in a screw conveyor type of cooler, such as the Carbondale scraper chiller. If desired, of course, other types of coolers can be used such as a tubular heat exchanger or a jacketed kettle or drum with cooling water circulating through a jacket.

A standing or storage kettle may also be used, if desired, either to permit the fresh grease product to remain quiescent for a required period during which it changes from a thick solution of oil in soap and acquires the desired grease structure, or this kettle may be used merely as a temporary storage vessel.

For the production of aluminum soap greases it is desirable to use both the chiller and the standing kettle so that the hot grease may be quickly cooled down to the grease structure-forming temperature, then permitted to remain in a quiescent condition at that temperature until the desired grease structure has been acquired. If desired, two or more of these standing kettles may be used in order to provide for uninterrupted or continuous manufacture. It is also possible to discharge the grease from the chiller directly into shipping containers such as barrels, kegs or cans.

If desired, the whole equipment may be maintained and operated under a pressure slightly above atmospheric in order to prevent evaporation of water during the manufacture of a lime soap grease.

The invention will be better understood from a consideration of the accompanying drawing which shows a schematic lay-out of the equipment used in applying this invention to the manufacture of aluminum or lithium soap grease.

Referring to the drawing, aluminum soap 1, preferably in the form of a dry powder, is fed along with oil from a supply tank (not shown) through line 2 into the roller mill 3 where the soap and oil are mixed together in the form of a thick slurry and then passed by line 4 into the slurry supply tank 5 through slurry-metering pump 6 into line 7 where it meets oil being fed from line 2 through line 8 and oil-metering pump 9, and the mixture of slurry and oil passes from line 7 into orifice mixer 10 through line 11 into pipe coil heater 12 where the mixture is heated to the required grease blending temperature, and thence passes by line 13 into surge tank 14 and is then pumped by pump 15 through line 16 into the Carbondale chiller 17, into the standing kettle 18 where it is permitted to remain quiescent until the desired grease structure has been acquired, and finally through filter 19 and discharged into suitable containers (not shown) for shipping or storage. Suitable lines 20, 21, and 22 may be used for by-passing the surge tank 13, the chiller 17, and the kettle 18, respectively, if desired.

The advantages of this invention will be further apparent from a consideration of the following examples, showing the manufacture of an aluminum soap grease according to this invention.

Example 1

A blend of 33% aluminum stearate and 67% of 70 S. S. U. at 210° F. lubricating oil was mixed by hand in a beaker. This was fed through a heating coil of copper tubing 20 feet long and ¼" I. D., immersed in oil at 400° F. The exit

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temperature was 320° F. One part of this concentrate was mixed continuously in a small centrifugal pump with 4 parts by weight of 200 S. S. U. at 210° F. lubricating oil. The final temperature was 250° F. The flow rate was about 1 lb. per minute. The stream was cooled by a steel coil 10 feet long and ¼" I. D., immersed in water at 180° F. On standing, a good pressure gun grease resulted.

Example 2

A suspension of 8% aluminum stearate in 70 S. S. U. at 210° F. lubricating oil was prepared by first mixing a concentrate of 33⅓% and diluting this in a kettle. This mixture was fed through a coil of 20 feet length and ½" I. D., immersed in a bath of fused sodium and potassium nitrates at 400° F. The feed rate was approximately 1 lb. per minute and the exit temperature was 325° F. This melted grease was then held in a surge tank holding 15 lbs. for the transit time of approximately 15 minutes, allowing air which had been trapped in the suspension to escape. This melted, de-aerated grease was then pumped through a cooler coil immersed in running water at 125° F. The exit temperature was 150° F. and the grease was stored in containers at this temperature until set. A satisfactory cup grease was produced. Lithium, calcium, barium, lead, zinc, soda or other soap may be similarly employed to make the respective types of grease, or soaps such as soda, lithium, barium, lime freshly prepared in a kettle or other equipment by direct saponification of fats or fatty acid with the respective alkalis may be fed directly into the mixing equipment for blending with oil cooling and the formation of greases.

A lithium soap lubricating grease preferably is quickly cooled to a grease structure-forming temperature of about 100° to 150° F. after which the grease may be passed through transition zones where it is permitted to remain quiescent until it acquires the desired chilled structure.

The present application is a continuation in part of application Number 461,144, filed October 7, 1942, now Patent 2,372,052, issued March 20, 1945.

It has now been found that further improvement in the above described process can be made by preheating the large bulk of oil which is used for diluting the soap-oil slurry which is made at a temperature substantially below grease cooking temperature. If, as preferred, the dilution oil is heated to a sufficiently elevated temperature it then becomes unnecessary to use the heater 12 shown in Figure 1. In other words, referring again to Figure 1, the present invention comprises essentially a transfer of the heater 12 from its position following the mixer 10 to a position immediately before or after the oil feed tank in line 8.

The invention will be better understood from a consideration of Figure 2 which shows one suitable arrangement of the equipment according to this new modification of the invention, as applied to the manufacture of aluminum soap grease for the sake of illustration.

Referring to Figure 2, aluminum soap which, preferably in the form of a dry powder, is fed along with oil from a supply tank through line 2 and valve 24 at a temperature between about room temperature and about 150° F. into the roller mill 3 where the soap and oil, together with minor amounts of other addition agents such as antioxidants, thickeners, etc., which may

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be fed in through line 23, are mixed together in the form of a thick slurry and then passed by line 4 into the slurry supply tank 5 through slurry-metering pump 6 into line 7 where it meets oil being fed from line 2 through valve 25, line 8, heater 26, where it is heated by means of steam, Dowtherm or direct fire to a temperature of about 300° to 600° F. (which is preferably high enough to avoid extra heating after compounding with the soap slurry), on through oil feed tank 27 and oil-metering pump 9, and the mixture of slurry and hot oil passes from line 7 into mixer 10 which may be an orifice mixer or any other suitable mixer such as a Lancaster disperser which gives high speed, efficient mixing, through line 11, and then either through heater 12 where the mixture is further heated to the required grease blending temperature, or bypassed through line 28, and then passes by line 13 into surge tank 14 and is then pumped by pump 15 into a suitable cooler 17, such as a Carbondale chiller or a short, coaxial, cylindrical rotary cooler, into the standing kettle 18 where it is permitted to remain quiescent until the desired grease structure has been acquired, and finally through filter 19 and discharged into suitable containers (not shown) for shipping or storage. Suitable lines 20, 21, and 22 may be used for by-passing the surge tank 13, the chiller 17, and the kettle 18, respectively, if desired. If desired, the positions of heater 26 and oil feed tank 27 may be interchanged. If desired a preheater may be used in the inlet end of line 2 to partially heat all of the oil at least to the temperature desired for the oil used in making the slurry.

The advantages of this modification of the invention will be found apparent from a consideration of the following example showing the manufacture of an aluminum soap grease according to this invention.

Example 3

A cold slurry was prepared from the following materials:

	Pounds
Aluminum stearate	30
Diisobutyl phenol	3
Polybutene solution ¹	1
Heavy oil ²	58

¹ 8% solution of 80,000 average molecular weight polybutene in Mid-Continent type mineral oil having a viscosity of about 240 seconds Saybolt at 100° F.

² Coastal type oil having a viscosity of about 200 seconds Saybolt at 210° F.

About 22 volumes of the resulting slurry which had a temperature of about 90° F., were mixed with about 78 volumes of a naphthenic type mineral oil having a viscosity of about 70 seconds Saybolt at 210° F. which had been preheated to a temperature of about 440° F. in a 600 pound steam heater, using a Lancaster disperser for effecting the blending. The resulting mixture had a temperature of about 345° F. It was then cooled in an externally and internally cooled rotary scraped chiller to 110° F. The product contained 7.5% aluminum stearate and was found to be an excellent pressure gun grease of 331 worked penetration (A. S. T. M. at 77° F.). The hot grease was slightly foamy but on cooling the foam disappeared. It could, if desired, be de-aerated and dried in a surge tank, but this was not necessary.

One advantage of this modification of the invention is that a high heat transfer efficiency, as high as 500 B. t. u. per hour-square foot °F., can be obtained by heating the mineral oil by

itself, whereas the heat transfer coefficient is much lower, even as low as 5 B. t. u. per hour-square foot °F. in the case of heating a grease base or slurry because the presence of the soap reduces the heat transfer rate.

This invention is not intended to be limited to the specific examples which have been given merely for the sake of illustration, but only by the appended claims in which it is intended to claim all novelty inherent in the invention as well as all modifications coming within the scope and spirit of the invention.

We claim:

1. Process for manufacturing a grease which comprises mixing a soap and a mineral base lubricating oil at a temperature substantially below the temperature at which said soap blends with said oil to form a grease, said temperature being above room temperature and not over 150° F. to thereby form a slurry of soap and oil, diluting said slurry with additional oil, preheated with all the heat required for the grease cooking, and blending to give the proportions of soap and oil desired in the finished grease, and cooling the resultant grease stock at least to a grease structure-forming temperature.

2. Process according to claim 1 in which the dilution oil is preheated to a temperature of about 300° to 600° F.

3. Process for manufacturing aluminum grease which comprises mixing dry aluminum soap and mineral oil which has been preheated to a temperature of about 100° F. to form a slurry having a temperature of about 80°-100° F., diluting said slurry with additional hot oil having a temperature of about 400° to 500° F. sufficient to give a grease cooking temperature after blending with the slurry, and using proportions of slurry and dilution oil to give the proportions of soap and oil desired in the finished grease, mixing to make a substantially homogeneous mixture, and cooling the resultant grease stock at least to a grease structure-forming temperature.

4. Process according to claim 3 in which the slurry contains about 15% to 35% by weight of aluminum soap and about 65% to 85% by weight of mineral oil.

5. Process according to claim 3 in which the final mixture of slurry and oil has a grease cooking temperature of about 250°-400° F. before cooling.

6. Process according to claim 1 applied to the manufacture of an aluminum soap grease having a finished soap content of about 5% to 10% by weight.

7. Process according to claim 1 applied to the manufacture of a lithium soap lubricating grease, and in which the finished grease is quickly cooled to a grease structure-forming temperature of about 100°-150° F.

8. Continuous process for manufacturing lubricating grease which comprises continuously mixing a soap and a mineral lubricating oil base stock in concentrated slurry forming proportions at a temperature substantially below the temperature required for blending said soap and said oil stock to form a grease, said temperature being above room temperature and not more than 150° F., effecting said mixing in a mixer adapted to break up lumps efficiently and to make a homogeneous slurry, passing said slurry continuously into a slurry supply tank, continuously withdrawing in controlled grease forming proportions slurry from said slurry supply tank and hot dilution oil to give the proportions of soap and oil desired in the finished grease, continuously mixing said slurry and hot oil to make a substantially homogeneous mixture, in which that said resultant mixture has been heated to a temperature sufficient to effect complete blending of said soap and oil to form a grease, and continuously cooling the resultant grease stock at least to a grease structure-forming temperature.

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