Aug. 28, 1956 P. J. BAKER 2,760,936

MANUFACTURE OF LITHIUM GREASE

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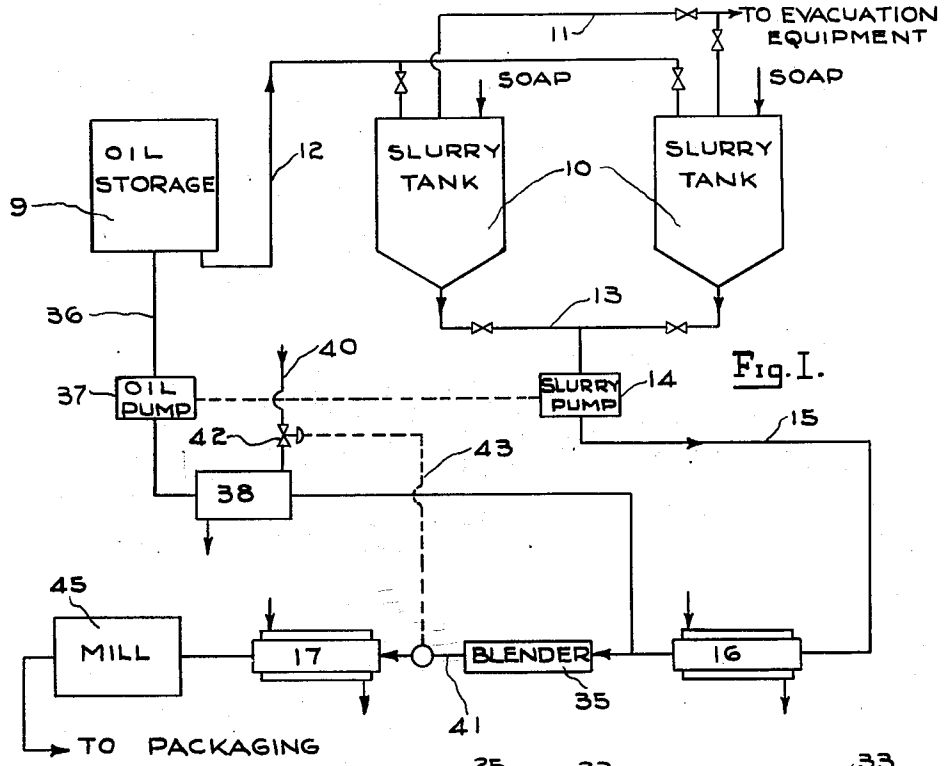


Fig. 1.

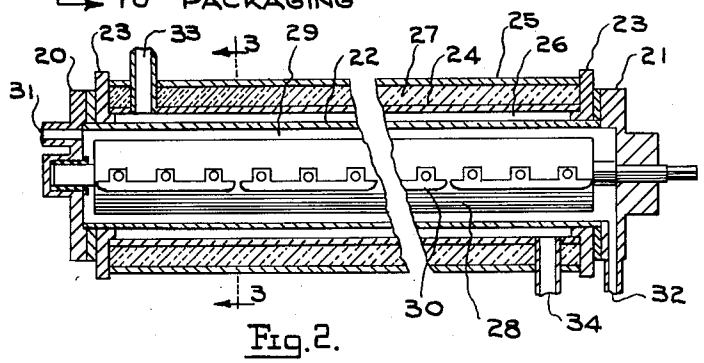


Fig. 2.

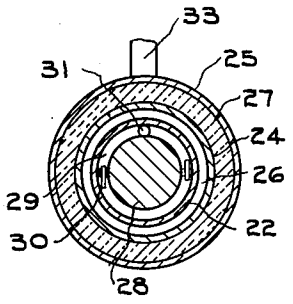


Fig 3

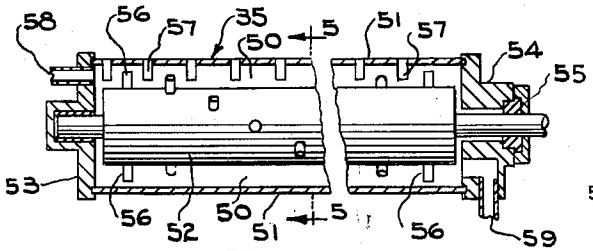


Fig. 4.

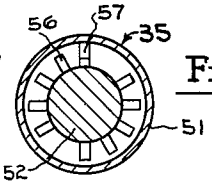


Fig. 5.

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Fig. 6.

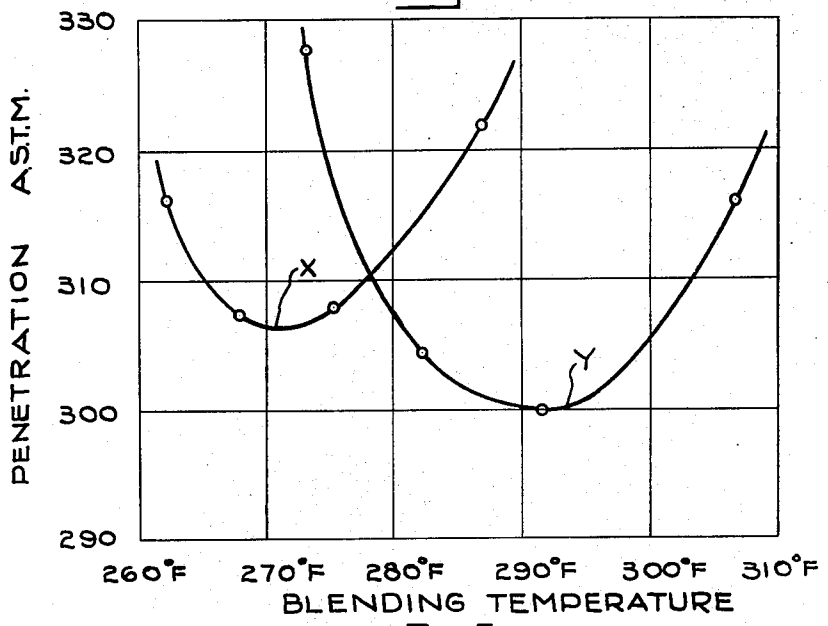
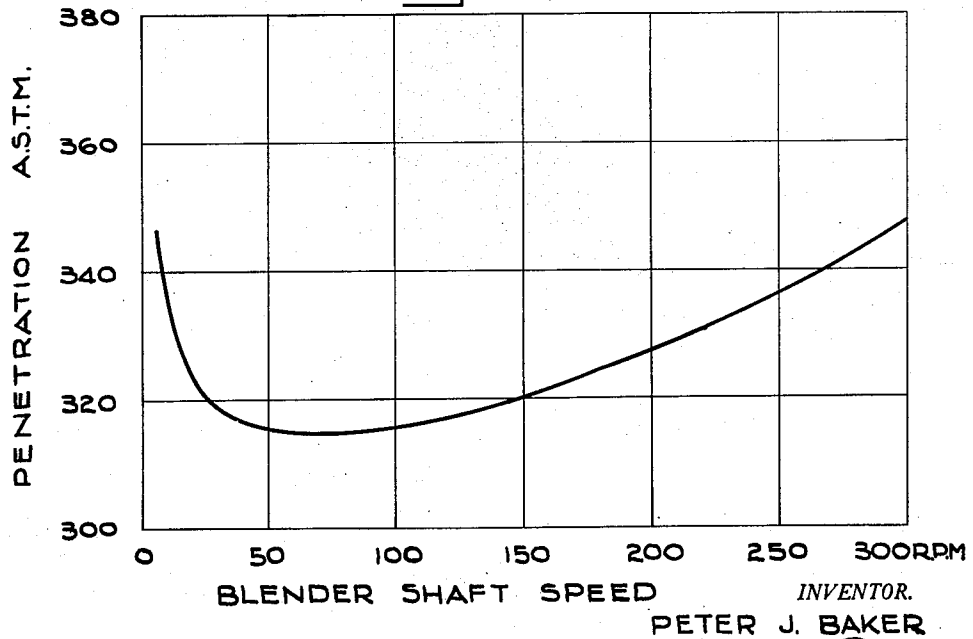


Fig. 7.



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2,760,936

MANUFACTURE OF LITHIUM GREASE

Peter J. Baker, Louisville, Ky., assignor, by mesne assignments, to National Cylinder Gas Company, Chicago, Ill., a corporation of Delaware

Application May 20, 1952, Serial No. 288,854

3 Claims. (Cl. 252—41)

This invention relates generally to the manufacture of grease compositions and more particularly to a novel continuous process for forming improved grease compositions of lithium soaps and oils.

It is well established that the quality of a grease varies substantially with the method of making it, and also that a satisfactory process for forming a grease of one metal soap and oil is not necessarily satisfactory, or even operative, for another metal soap even though the same oil be used. This is believed to be due to the fact that different types and lengths of fibers or micelle structures are formed by different soaps, and by the same soap when different processes of forming the fibers are utilized. Moreover, the degree of orientation and twist of the fibers is a variable from grease to grease and affects the physical properties thereof.

Grease fiber or micelle formation is essentially a process of crystallization which is brought about by cooling a solution of soap from a relatively high temperature to a lower temperature at which the soap is insoluble in the oil. Various factors including rate of cooling and the degree of agitation during cooling through the critical crystallization range and thereafter affect the type of fibers which are formed. After the fibers have been formed the structure may be modified by milling which tends to minimize sineresis and improve the stability of the grease.

The heretofore prevalent process for manufacturing lithium grease involves the following steps: Initially a slurry of lithium soap in mineral oil is prepared in open kettles and heated to a temperature of about 400° F. to completely dissolve the soap. The soap may be prepared separately or in the slurry kettles from fats or fatty acids and lithium hydroxide in the presence of part or all of the oil. If only part of the oil is used to form the soap the remainder of the oil is added after saponification is completed and the slurry is heated to solution temperature. The solution of lithium soap in oil is then drawn from the kettle and allowed to cool statically, usually in open pans, until a gel is formed. The cooled gel is then subjected to a milling operation in which it is worked to form a homogeneous grease composition. This process has a number of practical disadvantages. The high temperature heating in open kettles results in loss of more volatile components from the oil and creates a fire hazard. The process involves considerable manual handling of pans of gelled grease both in filling and feeding the gelled grease into the mill, which is time consuming and involves a danger of contamination. For these and other reasons, the process cannot be controlled to produce uniform greases from batch to batch, and, even more important, the yield of grease whish may be obtained from a given amount of soap is low. It is necessary to deaerate the grease during the milling operation. Furthermore from the standpoint of economy of operation, the power required to mill the cooled grease is quite high, and the heat requirement to raise the slurry including

the entire amount of oil in the grease to solution temperature is considerable.

I am aware that other processes have been proposed for the continuous manufacture of grease which in some respects more nearly resemble my new process than does the above described pan cooling method. Most of these other processes, however, are not specific to greases made from lithium soap and although they may work out satisfactorily for other metal soap greases they have not proved satisfactory for lithium greases, as evidenced by the continued use of the laborious pan cooling process by lithium grease producers.

I have provided an improved process for producing lithium grease which may be operated on a continuous or semi-continuous basis and which produces higher yields of grease from given amounts of soap (the most expensive constituent) and also produces grease of more uniform characteristics and superior quality. Moreover, my new process results in economies in labor, electric power and heat required.

One of the important features of my invention is the discovery that the above mentioned improvement may be obtained by blending a hot solution of the soap in part of the oil with additional oil having a substantially lower temperature, such blending being accomplished within a certain critical temperature range and under conditions of extremely mild agitation conducive to complete mixing with low shear. For practical purposes the critical temperature range lies between about 230° F. and 310° F. Following this rapid shock chilling by blending, the resultant grease is additionally cooled under conditions of violent agitation to a temperature below a second critical temperature below which the soap micelle is stable and which I have found to be about 160° F. The grease may then be finished by milling in the conventional manner. In connection with the milling operation, I have found that considerably less power is required to mill the grease than in the case of pan cooled greases. The reason for this is not fully understood, but it is believed to be due to the fact that the rapid chilling without substantial agitation during the blending step produces more soap micelle or fibers of the desired size and a lesser number of fibers of excessive length so that substantially all of the fibers are of the desired size and less milling power is required to break down overly long fibers.

This factor of proper fiber growth initially in the cooling step is also believed to account for the higher yields experienced with my process since the fibers which are formed are utilized with greater effectiveness in the gel to produce adequate penetration values or stiffness in the finished grease even though the soap is present in smaller amount. Obviously if a pan cooled grease must be worked to reduce extra long fibers, that same working reduces the shorter fibers to less than optimum gel forming length.

Another particular advantage of my process is that virtually no gel forming time is involved. Gelation is completed by the time the grease is cooled below the second critical temperature, and consequently no holding vessels are required.

Other objects and advantages of the improved process of my invention will become apparent to those familiar with the art on reading the following specification in connection with the drawings and the appended claims. In the drawings:

Fig. 1 is a flow diagram showing a preferred system of apparatus for carrying out my novel process;

Fig. 2 is a longitudinal vertical section showing somewhat schematically the internal construction of a preferred form of heat exchange unit;

Fig. 3 is a transverse section of such unit, taken on line 3—3 of Fig. 2;

Fig. 4 is a longitudinal vertical section showing somewhat schematically the internal construction of a preferred form of blender;

Fig. 5 is a transverse section of the blender taken on line 5—5 of Fig. 4;

Fig. 6 is a graph showing the effect of blending temperature on penetration; and

Fig. 7 is a graph showing the effect of blender shaft speed on penetration.

As previously indicated my improved process may be operated either continuously or semi-continuously. For continuous operation a pair of identical slurry tanks **10** are preferably employed. These tanks are provided with conventional agitators to keep the solids in suspension and are connected through a suitable line **11** to evacuating equipment (not shown) so that the slurry may be deaerated at this stage rather than in the finishing mill. Oil is supplied to the slurry tanks from the oil storage container **9** through conduit **12**, either by gravity feed or a pump (not shown). The soap is preferably previously prepared and added in solid form.

From the tanks **10**, which are discharged alternately, the slurry is conducted by a valved conduit **13** to a slurry pump **14** which forces it through the heat exchange and blending apparatus described below. The outlet of the pump **14** is connected by a conduit **15** to the slurry heating unit **16** where the slurry is agitated and heated to a solution temperature within the range of about 375° F. to about 450° F., depending upon the particular soap used.

The construction of heating unit **16** is preferably identical to that of the cooling unit **17**, so that Figs. 2 and 3 are illustrative of either of these two heat exchangers. Each unit comprises a pair of generally circular end plates **20** and **21** recessed to receive the ends of the heat transfer tube **22**. Ring members **23** are sleeved on the tube **22** adjacent to the end plates and support a pair of cylindrical elements **24** and **25**. Thus an annular space **26** is provided for heat transfer fluid around the tube **22**. The space between the sleeves **24** and **25** is preferably filled with insulation **27** to provide a heat insulating jacket.

The end plates **20** and **21** are provided with appropriate bearings and seals for journalling the agitator shaft **28** and sealing the point at which the shaft passes through the plate **21**. The agitator shaft **28** fills a major portion of the space within the tube **22**, and together with the tube forms a thin elongated annular passage **29** for the grease or slurry, as the case may be. The agitator shaft carries a plurality of blades or scrapers **30** which serve to prevent sticking of material to the inner surface of heat transfer tube **22** and to agitate the grease or slurry as the shaft rotates at relatively high speed, i. e. on the order of 500 R. P. M. A suitable drive mechanism (not shown) is provided to rotate the shaft at this speed.

In the case of the slurry being heated in the unit **16** the violent agitation aids in providing uniform heating and solution of the soap, while in the case of the grease being cooled in the cooling unit **17** the agitation serves to control micelle fiber growth and gel formation by promoting rapid controlled cooling from the critical blending temperature to the lower critical milling temperature.

The slurry or grease, as the case may be, enters the heat exchanger through an inlet conduit **31** at one end and leaves through an outlet conduit **32** at the opposite end. The heat transfer fluid is conducted to and from the annular space **26** by appropriate connections **33** and **34**.

To those familiar with the art of heat transfer it will be readily apparent that the units **16** and **17** effect very rapid and efficient heating and cooling of the slurry and grease under closely controlled conditions of temperature, pressure and degree of agitation. The heat transfer fluid in the case of the unit **16** is preferably a heating medium having a boiling point at atmospheric pressure much higher than that of water, such as diphenyl or diphenyl oxide or a mixture of the two sold under the trade name Dowtherm. Cold water or brine may be used in the cooling unit **17**.

Intermediate the two heat exchange units **16** and **17** a blender **35** is provided. A preferred form of blender is illustrated in Figs. 4 and 5. This unit is similar in construction to the two heat exchange units in that an annular passage **50** for the product is provided between a hollow cylinder **51** and a rotating shaft **52**. The ends of the cylinder **51** are supported by end plates **53** and **54** which contain suitable bearings for the ends of the shaft **52**. A seal **55** is provided in the plate **54** to prevent product leakage at the drive end of the shaft. Suitable means (not shown) are provided to drive the shaft at a slow speed to provide gentle agitation. Thorough mixing with low shear is accomplished by a series of pins **56** arranged in a helical pattern on the shaft **53** which cooperate with a row of similar stationary pins **57** mounted in the wall of the cylinder **51**. The product enters and leaves the blender through inlet and outlet connections **58** and **59**, respectively, provided in the end plates **53** and **54**.

The purpose of the blender, as will be explained later in more detail, is to mix the hot solution emerging from heating unit **16** with relatively cold oil. The blender alternatively may be in the form of a length of pipe having several bends therein or of a chamber having circuitous passages therethrough. The use of a slowly driven mixing shaft in the blender is preferred, however, because it imposes less load on the pumps **14** and **37** than does a static system. In the illustrated embodiment the oil is pumped from the tank **9** through a conduit **36** by means of an oil pump **37**. The oil pump and the slurry pump are preferably interconnected in any suitable manner to proportion the relative amounts of slurry and cold oil which are supplied to the blender. The ratio of oil in the slurry to cold oil is preferably about 1:1 although other ratios within the range of 3:1 and 1:3 have been tried and proved successful. In certain cases the oil from which the slurry is formed and the cold oil subsequently added may be of different types, in which cases separate oil storage tanks are used.

An oil heater **38** is shown in the drawing. This heater is not essential to the operation of the process, but its use is preferred for it provides a very simple arrangement for controlling the critical blending temperature. The heater **38** may be a conventional heat exchanger to which steam is supplied through a line **40**. The steam line **40** is preferably provided with an automatic control valve **42** which is connected as illustrated at **43** so as to be responsive to the temperature of the grease emerging from the blender **35** in the conduit **41** leading to the cooler **17**. Thus the critical blending temperature is controlled by supplying more or less heat to the relative cold oil prior to its being mixed with the hot solution in the blender **35**. The output temperature of the oil heater **38** is usually maintained within the range of 100° F. to 180° F., but actually this temperature is dependent upon the oil-slurry ratio and the blending and solution temperatures and may be varied considerably either above or below this range. For example, if a large proportion of the oil is initially incorporated in the slurry in slurry tank **10**, it may be necessary to refrigerate the cold oil to reduce the slurry from solution temperature to the desired blending temperature. In this latter situation the apparatus **38** takes the form of a refrigerating device rather than a heater.

The critical blending temperature may also be controlled by varying the relative amounts of oil and slurry or by varying the temperature to which the slurry is heated. The control of relative rates of oil flow, however, has proved much more difficult than the control of steam supplied to a heat exchanger such as indicated at **38**.

From the blender **35** the grease, at the critical blending temperature and in a "gutty" state indicating fiber or gel formation, is supplied to the cooling unit **17**. In this unit its temperature is quickly lowered under controlled agitation in the thin annular space **29** to below the critical milling temperature of about 160° F. At this temperature gel formation is believed to be complete and the grease is ready for milling and packaging. It is desirable not to

exceed the critical milling temperature in the milling operation, and, if the type mill employed produces considerable temperature rise, it is preferred to chill the grease to a somewhat lower temperature in the unit **17**. The milling operation, which may be performed in a suitable milling device indicated at **45**, differs from the conventional procedure in that deaeration is not required and in that considerably less milling power is needed as described. Experiment has shown that sufficient power is saved in the mill to more than offset the mechanical power used in the two heat exchange units **16** and **17** and in the blender **35**. Thus an overall economy in power is achieved by my process.

I have found that the blending temperature has very important bearing on final quality and characteristics of the grease, and that the blending temperature of each grease is quite critical. The effect of the blending temperature upon the penetration is shown graphically in Fig. 4. Penetration is a measure of hardness of grease and indirectly a measure of the "yield" which may be obtained from a given soap content, the lower the penetration and the harder the grease and the better the "yield." Curve X represents a grease formed of a lithium soap and a mineral oil having a viscosity index of 60. Curve Y represents a grease formed of a similar soap having a slightly lower softening point and a different oil, having a viscosity index of 41.8. In each case the soap content was 7%. As will be apparent from Fig. 4, the optimum blending temperature is about 270° F. in the first case and about 290° F. in the second. It has been found that different oils and soaps and percentages of soap produce similar curves but different critical blending temperatures. Although the exact relationships are unpredictable, nevertheless the critical temperatures may be readily determined by simple and conventional penetration test procedures. In general, however, the critical blending temperature has been found to be between about 230° F. and 310° F.

I also have found that the degree of agitation during blending is quite important. In Fig. 7 the relationship between agitation during blending and penetration is illustrated. In obtaining this curve a shaft type blender such as is illustrated in Figs. 4 and 5 was used and the speed of the shaft **52** was varied. The penetration of the greases corresponding to various shaft speeds was measured. The product cylinder **51** of the blender **35** used in this test had a diameter of three inches and was 12 inches in length. The cylinder **51** has a single row of stationary pins **57** spaced an inch part. The shaft **52** was two inches in diameter and had a series of pins which extended through the shaft to protrude on each side. The shaft pins **53** were spaced about an inch apart along the shaft axis and were arranged in a helix. As will be seen from the curve of Fig. 7 the optimum shaft speed is about 60 R. P. M. in this blender **35**. With this speed there is obtained a complete mixing of solution and cold oil and rapid cooling of the soap solution to critical blending temperature. The gentle agitation produces little shear and there are virtually no disruptive forces developed which might cause micelle fiber breakdown. For purposes of the process herein described, the extent of agitation employed at this point should be a minimum consistent with thorough mixing of the hot solution with the cold oil. Any greater degree of agitation involves excessive shear producing a softer grease and decreasing the possible "yield." This explains the equivalency of a length of pipe or labyrinth to the blender unit **35**.

A very important advantage of my process is the fact that the total heat requirement is substantially reduced since a large amount of the oil is not heated to solution temperature, as in the conventional process herein before referred to, but is introduced relatively cold. This is important from an operating as well as an initial equipment cost standpoint. For example, where about 50% of the oil is introduced cold the capacity of the heat source supplying heating medium to the unit **16** need be only half as great as if all of the oil had to be heated as in the conventional process.

As stated previously it is preferred to use a ratio of 1:1 between cold oil and oil in the slurry. When this ratio is maintained, it is possible to use heating and cooling units of the same size. The heating unit **16** handles only one-half the flow of the cooling unit **17** due to the fact that the cold oil does not pass through the heating unit. However, the temperature rise in the heating unit is about twice as much as the temperature drop in the cooling unit with the result that units having the same heat transfer capacity may be used.

The following specific examples may prove helpful in fully understanding my invention:

Example A

Fourteen pounds of prepared lithium stearate known as Witco #306, having a softening point of 210° C. and manufactured by the Witco Chemical Company, 295 Madison Avenue, New York, New York, were mixed with eighty-six pounds of a naphthenic petroleum oil, having a pour point of −15° F., a flash point of 410° F., a viscosity of 650 SSU at 100° F. and 60.6 SSU at 210° F., and a viscosity index of 41.8, to form a slurry containing 14% lithium soap. This slurry was then pumped through a heating unit similar to unit **16** shown in Figs. 1–3 at a rate of approximately 75 pounds per hour. The shaft speed in the unit was 500 R. P. M., and the temperature of the slurry was raised to about 425° F. The resultant hot solution was blended with one hundred pounds of the same oil having a temperature of about 160° F. by pumping the oil and hot solution through a 10 foot length of ½ inch pipe having eight right angle bends to form grease at a temperature of approximately 290° F. The cold oil was supplied at a rate of about 75 pounds per hour so the combined flow through the pipe was about 150 pounds per hour. Pumping the hot solution and cold oil through the pipe produced a gentle agitation equivalent to that provided in the rotating shaft blender described above when the shaft is rotated at very low speeds to provide rapid chilling of the hot solution with very low shear. Immediately after blending the grease was passed through an agitated cooling unit, similar to that hereinbefore described and having an agitator shaft speed of 500 R. P. M., to lower its temperature to below 150° F. The rate of flow through the cooling unit was about 150 pounds per hour. Next the grease was worked in a Charlotte colloid mill (Model W10).

The finished grease had a soap content of 7% and a penetration (ASTM) of 300 after 60 strokes in a conventional grease worker. It exhibited no bleed upon standing and stood up well on additional working, showing a difference of penetration of only 33 after 10,000 strokes in the grease worker.

In order to determine the effect of blending temperature on the "yield" as measured by penetration, a series of greases was prepared by the procedure of the above example with all conditions maintained the same except that the cold oil temperature was varied to give a series of blending temperatures from about 274° F. to about 307° F. The 60 stroke penetrations of the resultant greases were measured and the results when plotted gave the curve Y illustrated in Fig. 6. The grease having the lowest penetration value was obtained with a blending temperature of about 290° F.

Example B

Fourteen pounds of a prepared lithium stearate known as Witco #305 and similar to the soap in Example A, but having a slightly lower softening point, namely 200° C., were added to eighty-six pounds of mineral oil having a flash point of 380° F., a pour point of 0° F., a viscosity of 567 SSU at 100° F., and a viscosity index of 60. The oil and soap were mixed cold to form a slurry having 14% soap. A stream of such slurry was continuously with-

drawn and heated to 425° F. by the procedure of Example A to dissolve the soap and then was blended with a stream of cold oil having various temperatures ranging from 120° F. to 160° F. in the blender 35 described in connection with Fig. 7. The two streams were supplied to the blender 35 at equivalent rates so that equal volumes of hot solution and cold oil were mixed. This produced blending temperatures ranging from 262° F. to 285° F. The blender shaft speed was about 60 R. P. M. The penetration values of the resultant greases, which were cooled and finished as in Example A, were measured and plotted to produce curve X of Fig. 4. As shown by this curve the optimum blending temperature for maximum "yield" occurs in the range of 268° F. to 275° F. for the particular oil and soap utilized. All of the resultant greases had good texture and appearance and were not subject to sineresis.

Example C

A slurry was prepared from the soap described in Example A and the oil referred to in Example B. This slurry contained 25% soap. Portions of this slurry were heated to approximately 425° F. and then were separately blended with sufficient cold oil in the mixing blender described in connection with Fig. 7 to produce greases having soap contents of 7%, 9.5% and 11%, respectively. The blender shaft speed was 60 R. P. M., and the blending temperature in each case was held at around 270° F. by adjusting the cold oil temperature which was varied between about 145° F. and about 210° F. After blending, the greases were cooled and milled as in Example A.

Although somewhat more viscous than the slurries of the preceding examples which had lower soap contents, the 25% slurry was easily pumped through the system by virtue of the fact that the rotary blender was employed. No significant differences in appearance, texture or stability between greases made by my process from 14% slurries and from 25% slurries were noted, and all of the greases of this example were free from syneresis. The penetration values of the three greases of this example were as follows:

Percent Soap in Grease	Penetration (ASTM) After Working in Grease Worker	
	60 Strokes	10,000 Strokes
11% Soap	287	320
9.5% Soap	310	325
7.0% Soap	322	350

Example D

Lithium hydroxide and stearic acid in combining proportions were mixed in a gas fired heating kettle with two thousand pounds of mineral oil to form a solution containing 14% soap after saponification was completed. The temperature of the mixture was raised to 400° F. to effect saponification of the acid and hydroxide and complete solution of the soap produced. The mineral oil used had a viscosity of 500 SSU at 100° F. Two thousand pounds of cold oil having a temperature of about 140° F. were rapidly run into the kettle to lower the temperature of the batch to about 270° F. During the foregoing steps the batch was stirred gently by conventional paddle agitators rotating at 30 R. P. M.

The resulting grease was then run through a chilling unit similar to the unit 17 and its temperature was rapidly lowered to about 120° F. The throughput rate was approximately 1800 pounds per hour and the agitator shaft speed was 400 R. P. M. The cooled grease was milled in a centrifugal grease worker and packaged.

The resultant grease was free from syneresis and exhibited good stiffness both before and after additional working in a conventional grease worker. The 60 stroke penetration (ASTM) was 234 and the penetration after 5000 strokes was 275.

From the foregoing specific examples, which are included for purposes of illustration and not by way of limitation, it will be apparent that my process may be carried out with various different types of apparatus and may also be varied to utilize different oils, soaps and slurry compositions.

Various other changes and modifications, in addition to those set forth herein, may be made without departing from the spirit of my invention the scope of which is commensurate with the following claims.

What is claimed is:

1. The process of preparing lithium grease comprising forming a slurry of lithium stearate in a portion of the mineral oil desired in the finished grease, continuously passing a stream of said slurry through a heating zone and heating the slurry in said zone to a temperature at which the lithium stearate is completely soluble in the oil and above about 400° F. while subjecting the slurry to violent agitation sufficient to produce a substantial shearing action, thereby to effect thorough and intimate mixing of the lithium stearate and oil and form a homogeneous solution of the lithium stearate in the oil free from undissolved lithium stearate particles, adding the remainder of the oil desired in the finished grease to the solution under conditions of substantially shear-free gentle agitation, the added oil having a temperature selected with regard to the proportionate amount of the oil added in this step to produce a resultant temperature of the mixed oil and solution of about 280° F., passing the resultant mixture through a cooling zone under conditions of violent agitation sufficient to produce substantial shearing action and rapidly cooling said mixture in said zone to a temperature below 160° F., and finally milling the cooled product for a sufficient time to eliminate syneresis and provide stability in the finished grease.

2. The process of preparing lithium grease comprising forming a slurry of lithium stearate and mineral oil containing substantially all the lithium stearate and approximately half the oil desired in the finished grease, raising the temperature of the slurry to a temperature consistent with complete solution of the lithium stearate in the oil and above about 400° F. to completely dissolve the lithium stearate in the oil and form a homogeneous solution free from undissolved lithium stearate particles by passing the slurry in a thin layer under agitation in contact with a heat transfer wall, continuously mixing the remainder of the oil desired in the finished grease with the hot solution under conditions conducive to mixing with a minimum of shear to provide intimate mixing without micelle disruption, the added oil having a temperature such as to produce a resultant temperature of the mixed oil and solution within the range from about 230° F. to 310° F., passing the resultant mixture through a cooling zone under conditions of agitation to reduce the temperature thereof below 160° F., and finally milling the product for a sufficient time at a temperature below 160° F. to eliminate syneresis and produce stability in the finished grease.

3. The process of preparing lithium grease comprising forming a slurry of lithium stearate and mineral oil containing substantially all the lithium stearate and a portion of the oil desired in the finished grease, raising the temperature of the slurry to a temperature consistent with complete solution of the lithium stearate in the oil and above about 400° F. to completely dissolve the lithium stearate in the oil, adding the remainder of the oil desired in the finished grease under mild substantially shear-free agitation, the added oil having a temperature closer to room temperature than to the solution temperature and selected with regard to the proportionate amount of the oil added in this step to produce a resultant temperature of the mixed oil and slurry of between about 230° F. and 310° F., passing the resultant mixture through a cooling zone in contact with a heat transfer wall under conditions of agitation to reduce the temperature thereof below 160°

F., and finally milling the resultant grease at a temperature below 160° F. for sufficient time to eliminate syneresis.

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