

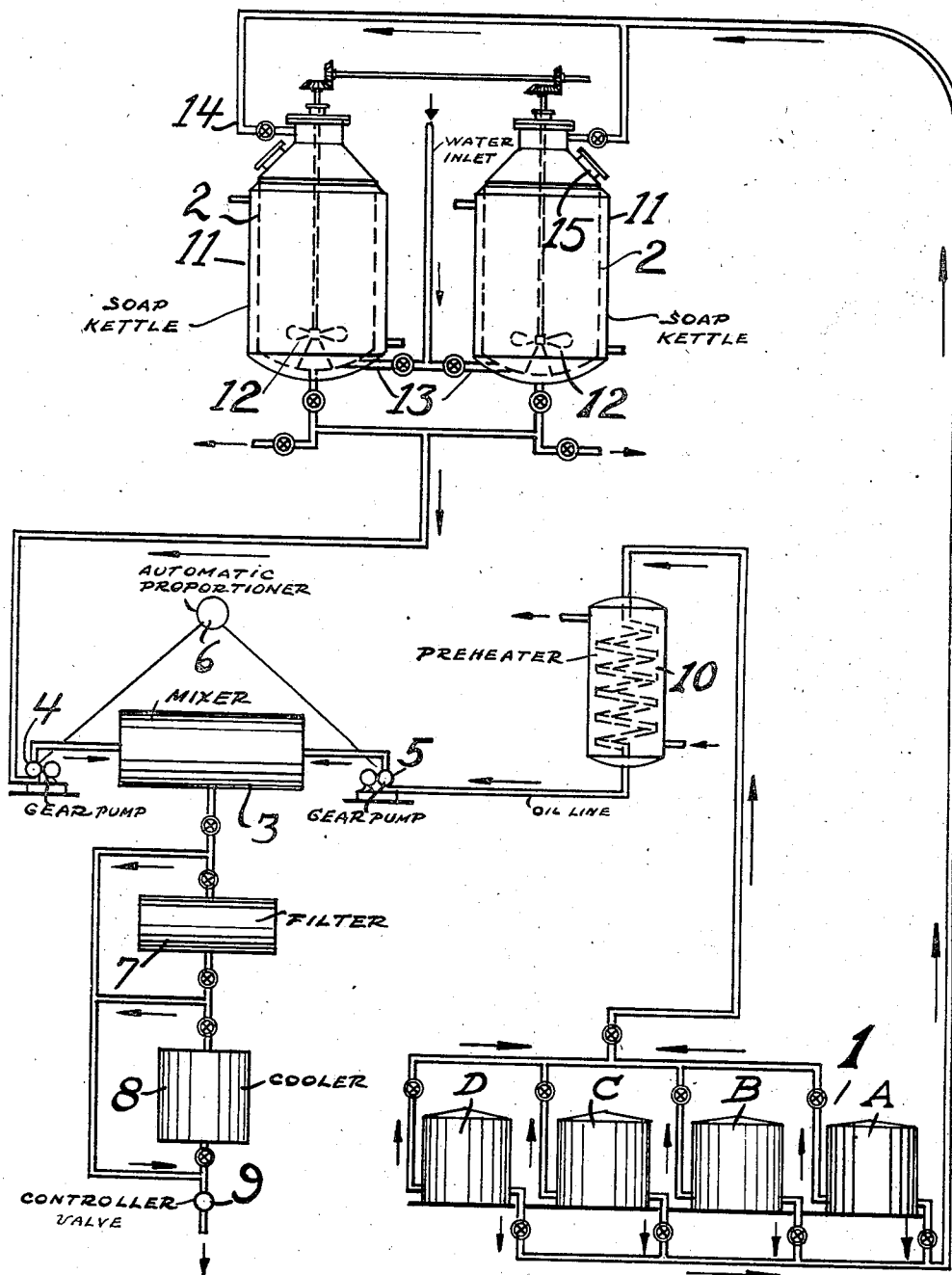
May 11, 1943.

A. E. CALKINS

2,318,668

GREASE MANUFACTURE

Filed June 24, 1940



Austin E. Calkins Inventor
By *R. Young* Attorney

UNITED STATES PATENT OFFICE

2,318,668

GREASE MANUFACTURE

Austin E. Calkins, Westfield, N. J., assignor to
Standard Oil Development Company, a corpora-
tion of Delaware

Application June 24, 1940, Serial No. 342,081

14 Claims. (Cl. 252—39)

This invention relates to a new process for manufacturing lubricating greases, and to the products obtained thereby, as well as the particular type of equipment used in making these greases. The invention relates more particularly to a continuous process for blending a preformed soap stock in various proportions with mineral lubricating oils of the desired viscosity, in order to produce various types and grades of lubricating greases.

There have been several attempts in the past to develop continuous processes for manufacturing lubricating greases but for various reasons these have not met with the desired success. For instance, it has been suggested to feed a finely divided soap in solid form and a mineral oil into a high-speed centrifugal pin disc mill, but satisfactory dispersion is not obtained because that procedure is not even recommended for lime soap greases containing 15% or less of soap in the finished grease, and even the greases containing the recommended range of 16% to 24% of lime soap must be kept boiling for 15 to 30 minutes or longer after it has been discharged from the mill. Another proposal was to continuously feed a solution of fatty oil in hydrocarbon oil through a series of tubes in a furnace heated with a suitable burner, and then continuously inject alkali into that heated oil mixture at various points during its progress through the furnace. This procedure has several disadvantages including that when the fatty oil or fatty acid is diluted with all of the mineral oil to be used in making the grease, slow and poor saponification is obtained, unless excessively high temperatures are used and that very poor mixing is obtained in the tubes with resultant danger of burning and uneven saponification. Other suggestions involve the actual preparation of a grease and then subjecting the latter to some treatment such as in a colloid mill, in order to homogenize it, and to remove any lumps of unevenly dispersed soap. Apparently the only field in which continuous operation has met with any degree of success is the field of lime sett greases, otherwise commonly called axle greases. Here, a suspension of lime in part of the oil is quickly mixed with a solution of rosin oil in the rest of the mineral oil, and then automatically filled into the grease cans, where saponification is allowed to complete itself after the grease mixture has been poured into the cans and permitted to stand. This procedure is satisfactory for the preparation of cheap, relatively low quality greases but is not suitable for higher quality products, chiefly because a substantial excess of lime must be used in order to get a reasonably quick saponification; also, greases made by this procedure, have a large breakdown in consistency upon working.

The chief reason for this lack of success in the

past is that the art of making good grease is fraught with many difficulties and uncertainties. Apparently, in order to obtain a satisfactory grease structure, (which in itself is somewhat difficult to define), a number of very sensitive factors must be controlled precisely; in fact, it seems to be well recognized that years of actual experience are necessary in order to make high-quality greases and that this art cannot be learned simply from studying instructions from a book. That is primarily why, commercially, practically all high quality greases are made by the batch process.

The primary object of the present invention is to develop a continuous process for making finished greases, by which advantage can be taken of the experience acquired by the grease operator in making a satisfactory soap stock for use in the grease, but whereby a more foolproof method is used for blending the soap stock with the required proportion and type of mineral lubricating oil to make the finished grease of the desired type. Another object of this invention is to develop a process for manufacturing greases such as to develop a grease manufacturing process, which can be carried out with simple and very compact equipment, without the requirement of many large blending tanks or kettles. Another object is to develop a grease-making process which requires substantially lower equipment installation cost than processes used heretofore, and at the same time to develop a process which will manufacture high-quality lubricating greases more economically than could be done in the past. These and other objects and advantages of the invention will appear more clearly from the following specification.

Broadly, the invention comprises a continuous process of grease making by continuously, and preferably automatically, mixing together proportioned amounts of heated preformed soap stock, preferably in the liquid state, and a heated mineral oil.

The soap stock may be prepared by any known method. For instance, the desired fat or fatty acid may be dissolved in the mineral oil, heated to the desired temperature and then saponified by adding the desired metal hydroxide or other suitable derivative. (In some cases the oxide may be used.)

Another procedure is to mix lime and fat (with a small amount of water if desired) and heat the mixture until saponification has been completed, at which time a small amount of mineral oil may be added if desired in order to make the soap stock more fluid at the temperature at which it is desired to be blended in later for making a finished grease.

A third alternative is to make one mixture of a portion of mineral oil with the fatty acid or fat to be used, and make another mixture com-

prising a suspension of the hydrated lime or caustic soda in another portion of mineral oil and then mix these two mixtures together, maintaining the desired temperature until saponification has been completed.

A still different procedure, and one which is preferred for the purposes of the present invention, is to mix the fat or fatty acid together with the lime or other saponifying agent, a small amount of mineral oil and water (if any is to be used), all together in a steam-jacketed pressure kettle, bolt on the lid and then heat to 250 to 325° F., or sufficiently to effect complete saponification, maintaining a slight superatmospheric pressure such as about 35 to 75 lbs. gauge. Pressure is used during the manufacture of the soap stock to accelerate saponification because it is desired to maintain the grease under pressure throughout the subsequent blending with mineral oil, until the grease is ready to be discharged into shipping packages.

In making a lime soap stock by the preferred procedure, the mixture of raw materials is heated to about 300° F., and cooked for 1 or 2 hours until saponification is completed; this is judged by examining a small sample of the product from time to time and examining its consistency or analyzing it.

In preparing the soap stock, any desired saponifiable material can be used, but it is preferred to use those containing fatty acid radicals of at least 10 carbon atoms. Either natural or synthetic materials can be used and either single materials or mixtures blended in order to obtain modified properties in the soap stock can be used. Suitable oils, fats, and fatty acids include those of animal, vegetable and fish origin, although the animal type are preferred. These fatty materials may be either saturated or slightly unsaturated but preferably not of the drying oil type. Besides the most common lard and tallow, examples of suitable materials include beef tallow, bone fat, castor oil, corn oil, cottonseed oil, cottonseed stearin, horse fat, lard oil, animal or hog fat, and wool fat or mixtures thereof. Examples, of suitable fatty acids include preferably those having 15 to 20 carbon atoms, oleic acid, stearic acid, animal fatty acids, cottonseed fatty acids, petroleum naphthenic acids and various synthetic fatty acids such as those obtained by oxidation of paraffin wax. Mixtures of fats and fatty acids may be used. Whether a fat or a fatty acid should be used, depends primarily upon whether glycerine is desired in the resulting soap stock because the saponification of a fat produces glycerine as a by-product. Also it is preferred to saponify the fat or fatty acids in the absence of mineral oil and then blend with mineral oil to a so-called soap stock.

Although the invention was primarily developed, and appeared to have outstanding advantages, for manufacturing calcium soap greases, other metal base greases may be used, for instance, sodium and aluminum, or mixed metal soap greases, e. g. sodium-calcium, or sodium-aluminum, etc. Such mixed greases are preferably made from mixed metal soap stocks, i. e. either two soaps are pre-mixed or they are made simultaneously by adding, for instance, both lime and soda to the material to be saponified. Greases may also be prepared from soaps of other metals such as potassium, lead, etc. although calcium, sodium, and aluminum are the most commonly used.

The composition of the soap stock to be used

for blending according to the present invention, will depend upon several factors, chief of which is the type of metal used in making the soap. For instance, a calcium soap stock should preferably contain about 45% to 70% or more soap, about 10% to 50% of oil, about 5% to 15% or even 50% or more of water, and 0% to 8% or so of glycerine. The sodium soap stock should preferably contain about 30% to 75% or soap, about 25% to 70% of oil, about 0% to 1% of water, and about 0% to 8% of glycerine. An aluminum soap stock will preferably contain about 15% to 50% of soap and about 50% to 85% of oil, usually no water, and perhaps 0% to 5% of glycerine, if desired. In general, the soap stocks therefore, will contain about 10% to 50% of oil and although this is preferred, it is possible under some circumstances to use a soap stock entirely free from oil provided it is maintained at a sufficient temperature to keep it in a fluid or readily soluble or dispersible state. The various percentages discussed above are by weight.

For more specific illustration of the soap stock composition which can be used, the following examples are given:

TABLE 1

Metal base	Per cent/weight				
	Soap	Oil	Water	Glycerin	Total
Calcium.....	65	22	8	5	100
Sodium.....	50	45	1	4	100
Aluminum.....	25	72	0	3	100

If any solid fillers are to be used in the finished grease, where the content of solid fillers may be from 0% to 40% by weight, such fillers are preferably incorporated into the soap stock during or after the preparation thereof. Some of the fillers which can be used include graphite, talc, asbestos, sulphur, etc.

Other materials, especially materials which have to be melted in order to be properly mixed, should preferably, if to be used at all, be mixed into the soap stock, for instance, oxidized asphalt, e. g. of 180 penetration, petrolatum, wax tailings, vegetable waxes, e. g. carnauba wax, etc. Such materials may be desirably used for preparing block greases, e. g. for open bearings or where the grease is applied as a lump.

Sufficient soap kettles of any conventional type equipped to meet the requirements of the process should be used to provide a constant supply of soap stock by timing the charges to the kettles so that one kettle of stock is always on hand completely finished, before another is exhausted so that a continuous supply of soap stock may be fed to the grease blending mixer.

The mineral oil to be used, referring either to the small amount which may be used in preparing the soap stock as stressed above, or the main bulk of oil to be blended with that soap stock as will be discussed hereinafter, may be any type of mineral oil stock normally used heretofore in batch operation for making the various types and grades of greases, for instance, mineral lubricating oil derived from various types of crudes, such as paraffinic, naphthenic, or mixed base crudes, may be used, as well as distillates or residual fractions thereof, with or without various treatments, such as clay treating, acid treating, extraction with selective solvents, such as liquid sulfur dioxide, phenol, etc., or lubricating oil fractions obtained by more or less syn-

thetic chemical processes, such as polymerization, condensation, hydrogenation, etc. Various natural or synthetic mixtures or blends may be used, for instance, one type of mineral oil may be used in making the concentrated soap stock and one or more other oils may be used in making the finished greases. The viscosity of the mineral oil to be used varies somewhat according to the type of metal used in making the soap. For instance, for calcium soap grease the blending oil should have a viscosity of about 35-100 seconds Saybolt at 210° F. but preferably about 40 to 80 seconds at 210° F. For a sodium soap grease the oil should have a viscosity of about 35-300 seconds at 210° F. but preferably 40-200 seconds at 210° F., and for an aluminum soap grease the oil should preferably have a viscosity of about 45-300 seconds Saybolt at 210° F. and preferably about 50-200 seconds at 210° F.

If any oil-soluble addition agents are to be incorporated in the finished greases, they should preferably be added to the oil stock. Various addition agents which may be added include, for instance, oil-soluble dyes (about 0.01% to 1% by weight on the finished grease), stringiness addition agents (0% to about 2% by weight), polyisobutylene, e. g. 1000-200,000 molecular weight, preferably above 30,000 molecular weight, e. g. 70,000 to 80,000 molecular weight, or natural or synthetic rubber; anti-oxidants (0.01 to about 0.3%), e. g. phenyl alpha naphthyl amine, glycols, phenols, phenolic ethers, amino phenols, alpha naphthol, etc.; and extreme pressure lubricating addition agents (0 to about 20% by weight) e. g. sulfurized fatty oils, chlorinated or sulfur-chlorinated hydrocarbons, etc., and various other materials commonly used in lubricating greases.

The oil stock to be blended for making the various greases should preferably be preheated, which can be accomplished readily by passing the oil through suitable heater or furnace, or by any other suitable method, as the oil stock is also preferably delivered under slight superatmospheric pressure, such as about 20 to 50 pounds so that the entire grease blending system will be under substantially even superatmospheric pressure. Pipe lines from supply tanks of various grades of mineral oil may be so selected and equipped with suitable valves so that any one oil, or a mixture of any two or more, may be used as desired.

The heated preformed soap stock and the heated mineral oil to be used for the blending are now ready to be mixed continuously according to the present invention. In order to explain this operation more easily, reference will be made to the accompanying drawing which is a schematic layout of suitable equipment for carrying out the invention.

Referring to the drawing, it is seen that the desired oil stock 1 obtained from one or more suitable sources such as A, B, C and D, is mixed with a soap stock prepared in soap kettle 2, by passing them through the mixer 3 after having been proportioned or metered out by the gear pump 4 for the soap stock and gear pump 5 for the oil stock. The feed of the pumps 4 and 5 may be controlled by hand if desired or preferably controlled by the automatic proportioner 6 which can be adjusted to give a wide range of proportions of the soap stock and the oil stock and at the same time it is so designed that with any particular proportion of soap and oil, the total capacity or throughput of both may be in-

creased without disturbing the proportions of each. The mixer 3 may be any one of a number of high speed mixers in which the materials to be mixed are brought into contact in a highly turbulent zone, whereby efficient dispersion is obtained by means of the shearing action of rapidly moving parts alone or combined with the directioning effect of one or more small vanes or baffles. The preferred type of mixer is a centrifugal pump or a mixer known in the industry as the Lancaster disperser. This latter machine consists essentially of a horizontal operating tube with duplex dispersing mechanism each consisting of two impellers attached to the shaft with a set of stator vanes held by tubed wall between impellers. The impellers of one mechanism are of opposite hand to those of the other set, so that the thrust of the two sets of impellers is therefore counterbalanced, practically eliminating end thrust load on the bearings. The radial length of the impeller blades is relatively small so that all the material processed is confined to a thin annular ring where it is subjected to a high average peripheral velocity.

Also other types of mixers include a centrifugal pump, or a plurality of such pumps arranged in series so as to obtain more mixing and a fine degree of dispersion, or other types such as a screw pump or screw conveyer, high speed mills, such as a colloid mill, roller or disc mill or paint mills, etc.

The grease issuing from the mixer 3 may then be passed through suitable filter 7 and if desired a cooler 8 and then, after going through a loaded pressure controller 9, is discharged or filled into grease cans for storage or shipment. As indicated in the drawing, by suitable pipe lines and valves the grease may be bypassed around the filter and cooler.

An example of the grease making operation is as follows:

Oil stock 1 may be heated to the desired temperature in a suitable preheater 10; and the soap kettle may be heated by any suitable means, such as by steam jacket 11 or by direct fire. The soap kettle 2 is also provided with suitable agitators 12 which may be driven by any suitable source of power not shown. The materials to be used in making the soap stock may be charged into the soap kettle in any desired manner. One way of doing this is to admit the water near the bottom of the kettle through a line 13, admit the small amount of mineral oil near the top of the kettle through the line 14 and charge the saponifiable material and metal compound, e. g. hydrated lime, caustic soda, etc., or a preformed soap, e. g. aluminum stearate or aluminum naphthenate, etc., through the charging openings 15. It is to be understood of course that the entire system will be provided with suitable pressure gauges, safety valves, thermometers and control valves, as needed for proper operation of the equipment according to the purview of the invention. If desired, the steam jacket around the soap kettle may be connected to water lines so that if the soap stock has been prepared, it may be cooled by passing water through the catalyst jacket.

The length of time during which the soap stock and oil stock are subjected to mixing will of course depend upon the type mixer used but will normally vary within the approximate limits of 5-30 seconds, and preferably from about 10-20 seconds.

As suggested previously, it is preferred to have the entire system including the oil feed lines, the

soap kettles, the proportioning pumps, and the mixers, including also the filter and cooler if such are used, under a slight superatmospheric pressure of, for instance, from about 15 to 50 lbs./sq. in. The primary reason for this is to prevent the evaporation of water when making calcium soap greases, because anhydrous calcium soap usually will not make a grease having a suitable grease structure. The water content should be about 5-25% (on the weight of dry soap present). Ten per cent would be theoretically correct but in practice a slight excess is desirable so that at least about 12% is the preferred amount. The use of superatmospheric pressure also assists in preventing foaming. The exact pressure to be used, as will be obvious to those skilled in the art, will depend upon the temperature to which the soap stock or the finished grease is heated, the higher these materials are heated, the higher the pressure will have to be in order to prevent evaporation of the water.

In carrying out the invention, it is critically important to maintain the proper temperatures in the soap kettles and in the oil stock so that the mixture thereof or the finished grease may be maintained within the proper temperature limits. The temperature of the oil stock should be approximately 125-450° F. in order to obtain the proper dispersion of the soap and preferably with lime soaps, about 175-210° F. so that the grease will have a temperature below 212° F. at the time of discharge. For aluminum soap greases, the oil stock temperature should be about 275-350° F. The soap temperature should be about 190-450° F. or even as low as 125° F. if the soap stock already contains a substantial amount of oil, or preferably about 190-300° F. for lime soaps, about 300-350° F. for aluminum soaps, and about 250-450° F. for soda greases. Lower temperatures tend to preclude proper solubility and dispersion, while excessive temperatures tend to cause burning and undesirable decomposition of some of the organic constituents in the soap stocks. The resultant grease temperature should be within the approximate limits of 150-450° F. and preferably about 175-210° F. for lime greases, about 250-350° F. for aluminum greases, and about 150-450° F. for soda greases. Especially when making a lime soap grease, temperatures below 175° F. tend to cause a decrease in the yield, by which term is meant the relation between the A. S. T. M. penetration of the grease for any particular soap content. The temperature of the oil stock and the soap stock must be balanced in order to obtain the abovementioned desired range of temperature in the finished grease.

The proportions in which the soap stock and the oil stock are to be blended in order to make the finished grease, depend of course upon the type and grade of grease desired and upon the type of metal soaps used, i. e. whether calcium, sodium or aluminum soap is used, the viscosity and other characteristics of the oil stock, and whether other materials are present, such as glycerine or finely-divided solid fillers. Usually, however, the amount of soap in the finished grease will be within the approximate limits of 1 to 55 (1% soap giving a fluid grease), but preferably between the limits of about 5-25%. As will appear more clearly from the later discussion of the experimental data, the present invention has special advantages for greases containing less than about 15% of soap, and this is particularly true of lime soap greases. In order to obtain the greases having the desired soap content, about 1 to 20

parts by volume of the heated oil stock are mixed with 1 part by volume of the heated preformed soap stock, although usually the proportions are somewhat narrower, namely, about 3 to 15 parts by volume of oil to 1 part by volume of soap stock. The blending or mixing may be accomplished either in one stage or in a plurality of stages as desired, for instance, if it is intended to make a grease with a very low soap content, such as a calcium soap content of about 5% in the finished grease, one might start with a soap stock containing 65% of calcium soap, and first mix 1 volume of that soap stock with 4 volumes of oil and then re-mix 1 volume of the resulting grease with about 3 volumes of oil. By this use of a plurality of stages for the mixing, a better conversion is obtained than can be done in a single stage when starting with a very concentrated soap stock and ending with a very dilute or low soap content grease.

When making aluminum soap greases, the practice followed heretofore may be used if desired, namely, pouring the freshly made aluminum soap into pans, e. g. 3 ft. x 4 ft. x 6 in., to permit formation of the desired structure, and then put back into the kettle and re-stirred.

The present invention has many advantages, some of which were noted previously. One advantage is that it enables the use of more compact equipment for the commercial manufacture of greases; for instance, it is estimated that 2 small kettles of 50 gallon capacity each, when used according to the present invention, are equivalent in capacity to a large 600 gallon kettle when used according to the standard batch methods used heretofore. Another advantage is that this invention results in a greater uniformity of product in commercial operation than has been possible heretofore. This uniformity evidences itself in the soap content, consistency, color and stability of the greases. Furthermore, by reason of the uniformity in soap content, consistency, etc., this invention permits the manufacture of greases, especially lime soap greases, with a lower soap content than were heretofore considered possible from a commercial point of view owing to the difficulty of making a uniform product by the prior art batch methods and owing to the difficulties of making a product having a suitable quality by any previously proposed continuous method.

In order to illustrate this economy of soap content, the following table is given showing the yield which can be reasonably expected for manufacturing lime soap greases according to the present invention. The "yield" figures are expressed as the maximum worked penetration for various soap contents.

TABLE 2

Per cent soap	Worked penetration, maximum
8	320.
10	285.
12	265 pref. 260.
14	240 pref. 235.
16	220 pref. 215.
18	200 pref. 195.
20	185 pref. 180.
22	170.
24	155.
26	145.
28	135.
30	120.

The above figures do not mean, of course, that lime soap greases having a higher worked pene-

tration cannot be made according to the present invention, if it is so desired, but these figures indicate that if the optimum water content is used, as indicated previously, and the operating conditions are controlled for the purpose of making greases having the lowest possible penetration for any particular soap content, the resultant greases will normally have a worked penetration below the figures indicated in the above table. For the sake of comparison, it might be noted that a test of samples of lime soap grease made by 5 of the large commercial producers of such products, showed that the lime soap greases containing 14% of lime soap had a worked penetration between the limits of about 260-350 and that lime soap greases having a content of 20% of soap had a worked penetration of 185-265. Obviously, therefore, since the soap is one of the most expensive ingredients in the finished grease, the present invention is a distinctly valuable contribution to the art by showing how it is possible to commercially manufacture a uniform grease having a substantially lower worked penetration than has been possible heretofore for any particular soap content.

It is apparent that if the penetration figures in Table 2 are plotted graphically on a chart for the various soap contents given, the corresponding maximum worked penetration for other percentages of soap can readily be determined by interpolation or extrapolation.

In addition to the advantages mentioned above, the grease products of this invention are also substantially free from air bubbles, lumps, graininess and foam, and do not have as much tendency to separate oil on standing as do products of an equivalent soap content made by prior art methods.

In order to illustrate more clearly the operation and advantages of the invention, the following examples and data are given:

A lime soap stock was prepared from the following amounts of materials:

Lard	parts by weight	3,000
Hydrated lime	do	432
Oil	do	1,194
Water	do	1375

¹About 100-150 more parts by weight of water were added during the saponification and cooked out.

The lime, water and fat were mixed at about 150° F. in an open top steam-jacketed kettle. The mixture was heated to boiling (about 212° F.) and cooked for 1 or 2 hours until saponification was complete. Then the oil was stirred in and the cooking continued until the resulting soap stock was completely homogeneous.

The lard used in this test had an iodine number of 53 and a saponification number of 190 and the mineral oil was a paraffinic lubricating oil stock having a viscosity of 40 seconds Saybolt at 210° F., 110 seconds at 100° F., with a viscosity index of 80 and a pour point of +15° F.

The composition of the resulting soap stock was as follows:

	Per cent
Soap	65.9
Oil	24.8
Water	3.5
Glycerine	5.8

By means of laboratory equipment comprising 2 gear pumps controlled by an automatic proportioner for metering the desired proportion of soap and oil, and a centrifugal pump for mixing the proportioned amount of soap and oil, 1 vol-

ume of the soap stock was mixed with 3.57 volumes of the oil, in accordance with the teachings of this invention, the soap stock and oil stock being fed continuously through the mixer for about ½ hour, 8 samples being taken out at approximately 3 minute intervals throughout the run. The following data were obtained:

Soap temperature average	°F	250
Oil temperature average	°F	205
Mixed grease temperature average	°F	185
Unworked penet. (A.S.T.M.)		160
Worked penetration (A.S.T.M.)		266
Water content	per cent	0.85
Soap content	do	15.7

The product was a buttery cup grease, very satisfactory in most respects, but owing to the fact that an open kettle had been used for the preparation of the soap stock, more water had been boiled out of the soap than had been expected and consequently the soap stock and the resulting grease had a slightly lower water content than is desirable for the best yield (penetration for a given soap content). In other words, if a somewhat larger amount of water had been used (about 12% on the weight of the soap) the worked penetration would have been about 210 or 215 instead of 266. The consistency of the product of this example corresponds to a No. 2 grease (according to the National Lubricating Grease Institute standards) and the throughput in this laboratory equipment was about 150 grams per minute or about 20 lbs. per hour. The pipe lines feeding into the soap stock gear pump and the oil gear pump were ¼ inch in diameter.

It is estimated that on a commercial scale in similar but larger equipment, two soap kettles each holding 50 gallons or about 400 lbs. of soap stock would be used and pipe lines of about 2 inches in diameter would be used, with a resulting throughput of about 15 lbs. per minute or about 900 to 1000 lbs. per hour.

In order to show what a high degree of uniformity was obtained even in this relatively crude experimental layout where of course the pipe lines, gear pumps and mixer were subject to some change in temperature even though they had been insulated to some extent and were heated with electrical resistance wires wrapped around them, the test results giving the penetration and the percentage of water and soap in the 8 samples taken throughout the run, are shown herebelow, together with a summary showing the maximum, minimum, and the difference for each property.

TABLE 3
#2 cup grease

Sample No.	Penetration (A. S. T. M.)		Per cent	
	Un- worked	Worked	Water	Soap
1	155	261	.90	15.9
2	152	262	.85	15.7
3	156	262	.95	15.3
4	158	267	.75	15.4
5	151	265	.85	16.0
6	163	273	.90	15.8
7	162	274	.80	15.9
8	157	263	.90	16.0
Aver	157	267	.87	15.75
Max	163	274	.95	16.0
Min	151	261	.75	15.3
Diff	12	13	.20	.7

The above table shows that in all of the 8 samples tested throughout the ½ hour of continuous

operation, the difference or spread between the maximum and minimum for the unworked penetration was only 12 points and for the worked penetration only 13 points (from 261 to 274), the water content only 0.20% and the soap 0.7% (from 15.3 to 16.0). It is expected that with commercial operation, even still better uniformity can be obtained, but even these results are exceptionally good compared to successive batch operations according to prior art methods.

A large number of other continuous runs were made, using either lime soap or soda soap stock and using various proportions of soap to oil and various rates of throughput. The average temperatures of the soap stock, the oil stock and the resulting grease, the throughput expressed in pounds per hour, the average soap content and worked penetration of the resulting grease, are all tabulated in the following Table 4; and the unworked and worked penetrations, as well as the water content and the soap content of the various individual samples taken during the continuous operation of each test, are shown in Table 5. The inspection of the several oils used as the oil stock in the various tests is shown in Table 6.

The above data show that when various types and grades of greases are produced according to the present invention, unexpectedly superior results are obtained in regard to economy of operation, uniformity of product, and production capacity with relatively small compact equipment.

It is not intended that this invention be limited to any of the particular examples which were given merely for the sake of illustration, nor by any theory as to the mechanism of the operation of the invention but only by the appended claims in which it is intended to claim all novelty inherent in the invention as broadly as the prior art permits.

I claim:

1. Continuous process of grease making which comprises continuously mixing together proportioned amounts of heated preformed soap stock and a heated oil in a high-speed mixer in which the soap stock and oil are brought into contact with each other in a highly turbulent zone; whereby efficient dispersion is obtained by means of the shearing action of rapidly moving parts.

2. Process according to claim 1 in which a calcium soap stock is used.

TABLE 4

Test No.	Soap stock (parts by weight)				Temperatures (average)			Average		
	Fat	Oil	Water	Lime	Soap	Oil	Grease	Through-out	Soap	Worked penetration
		<i>Lime soap stocks</i>			°F.	°F.	°F.	Lbs./hr.	Per cent	
2	60.00	23.76	7.60	8.64	244	208	170	12	15.3	228
3	65.4	17.6	7.6	9.4	238	200	183	15	17.8	187
4	64.2	20.9	5.6	9.3	254	212	170	16	15.7	214
5	65.0	17.65	8.0	9.35	236	214	183	18	9.7	281
6	65.0	17.65	8.0	9.35	238	220	192	18	9.5	311
7	65.0	17.65	8.0	9.35	259	207	170	15	10.0	273
		<i>Soda soap stocks</i>								
	Soap			Glycerine						
8	31.26	67.09	0.05	1.50	257	185	185	10	7.5	362
9	26.00	72.65	0.05	1.30	235	170	190	20	6.8	364
		<i>Aluminum soap stocks</i>								
10	33.30	66.70	0	0	305	350	290	10	7.9	315

TABLE 5

Test No.	Sample No.	Penetration		Composition of grease	
		Un-worked	Worked	Water	Soap
				Per cent	Per cent
2	1	139	234	1.90	14.5
	2	133	227	1.90	15.6
	3	127	228	1.95	15.0
	4	139	221	1.90	16.3
	Average	134	228	1.91	15.3
3	1	90	184	2.25	17.8
	2	94	184	2.00	18.6
	3	92	189	1.95	17.4
	4	92	189	2.00	18.0
	5	97	187	2.05	17.1
	6	99	187	2.05	17.1
	Average	94	187	2.05	17.8

TABLE 6

No of the tests in which this oil was used

	2-5, 7	6 ¹	8 and 9 ²	10
Vis./210	39.7	70.5	163.0	170.
V. L.	80	40	96	35.
Pour pt.	+25	0	+50	0.
Type of crude	Paraffinic	Naphthenic	Paraffinic	Naphthenic

¹ 11½% of polyisobutylene having a molecular weight of about 70,000 was dissolved in this oil.

² Pennsylvania oil having a viscosity of 165 seconds at 210° F.

3. Process according to claim 1 in which the soap stock contains some oil.

4. Continuous process of grease making which comprises continuously mixing together 1 volume of heated preformed soap stock containing 10% to 86% by weight of oil and 15% to 75% by weight of soap, and 1-20 volumes of heated mineral oil in a high-speed mixer in which the soap stock and oil are brought into contact with each other in a highly turbulent zone, whereby efficient dispersion is obtained by means of the shearing action of rapidly moving parts, combined with the directioning effect of a plurality of small vanes, and thereby continuously producing a finished grease containing 1% to 35% by weight of soap.

5. Process according to claim 4, in which the preheated soap stock is maintained at a temperature of 190° F. to 450° F., the preheated oil is maintained at 125° F. to 450° F. and the resultant grease is produced at a temperature of about 150° F. to 450° F.

6. Process according to claim 1, carried out under superatmospheric pressure.

7. Process according to claim 1, carried out under a superatmospheric pressure of about 15-50 lbs. per sq. in.

8. Process according to claim 1 in which the mixing is effected continuously in a highly turbulent zone by a high-speed mixer whereby effi-

cient and substantially uniform dispersion is obtained in a time not substantially exceeding 30 seconds.

9. Continuous process of grease making which comprises maintaining a continuous supply of calcium soap stock, continuously mixing together in a high-speed mixer having a dispersing action substantially equivalent to that of the Lancaster disperser, 1 volume of said soap stock at a temperature of about 190° F. to 300° F., and 1 to 20 volumes of mineral oil maintained at a temperature of 175° F. to 210° F., under a superatmospheric pressure sufficient to prevent vaporization of the water in the soap stock under the temperature used, thereby continuously producing a calcium soap grease at a temperature of about 175° F. to 210° F.

10. Process according to claim 9 in which the time of mixing is about 5-30 seconds.

11. Process according to claim 9 in which the soap stock contains 10% to 85% by weight of oil and about 15% to 75% by weight of calcium soap.

12. Continuous process of grease making which comprises continuously metering proportioned amounts of heated preformed soap stock in a semi-liquid state and a heated mineral oil by means of gear pumps regulated by an automatic proportioner, and continuously mixing said proportioned amount of soap stock and mineral oil together in a Lancaster disperser.

13. Continuous process of making a lime soap grease which comprises maintaining a continuous

supply of calcium soap stock having a temperature of about 190° F. to 300° F. and having substantially the following composition by weight:

Calcium soap-----	percent--	45-70
Oil-----	do-----	10-50
Water-----	do-----	5-50
Glycerine-----	do-----	0- 8

continuously mixing together in uniform controlled proportions one volume of said soap stock with 1 to 20 volumes of mineral oil maintained at a temperature of about 175° F. to 210° F., and having a viscosity of about 35-100 seconds Saybolt at 210° F., in a highly turbulent zone by a high speed mixer whereby efficient and substantially uniform dispersion of said soap stock and oil is obtained in a time not substantially exceeding 30 seconds, under a superatmospheric pressure of about 15-50 pounds per square inch sufficient to prevent vaporization of the water in the soap stock under the temperature used, thereby continuously producing a calcium soap grease having a temperature of about 175° F. to 210° F., and, without substantial further mixing, passing the finished grease through a pressure-reducing controller valve and drawing the grease into containers for storage and shipping.

14. Process according to claim 1 in which the time of actual mixing is not substantially more than 30 seconds.

AUSTIN E. CALKINS.