April 8, 1958 R. F. NELSON ET AL 2,830,022

METHOD OF GREASE MANUFACTURE WITH RECYCLE COOLING

Filed June 30, 1953 2 Sheets-Sheet 1

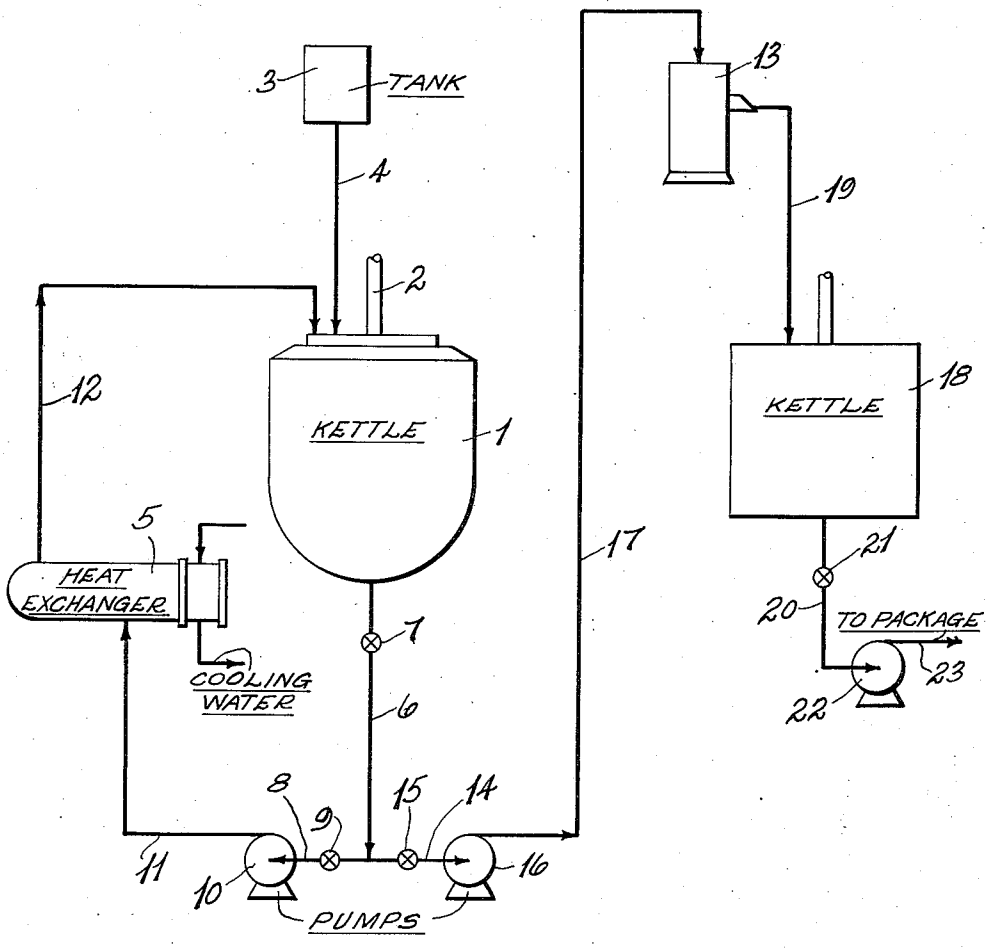


Fig. 1.

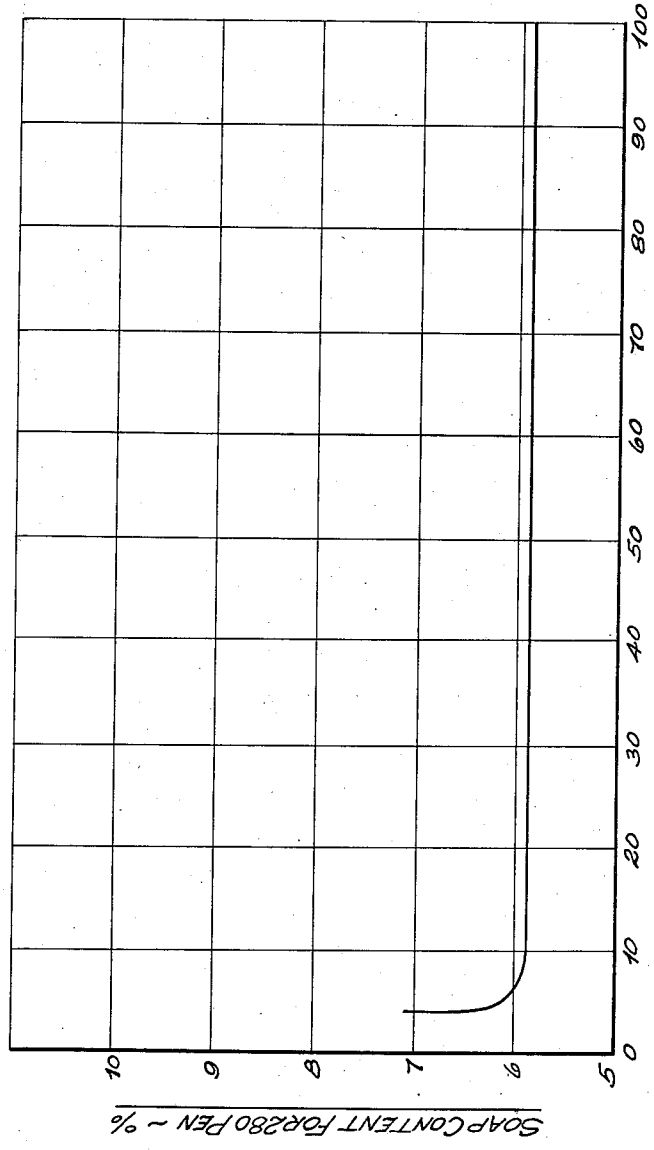
INVENTOR.

BY

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Filed June 30, 1953 2 Sheets-Sheet 2



EFFECT OF COOLING RATE ON YIELD OF LITHIUM 12-HYDROXYSTEARATE GREASE IN TEMPERATE RANGE OF 400-300°F.

Fig. 2.

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METHOD OF GREASE MANUFACTURE WITH RECYCLE COOLING

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7 Claims. (Cl. 252—41)

This invention relates to improvements in the manufacture of lubricating greases. More particularly, it relates to an improved process for the manufacture of greases containing lithium soaps of hydroxy fatty acids.

In accordance with this invention, greases are produced by a method which involves cooling a hot grease mixture down from a temperature above its gelation temperature until a grease consistency is obtained by continuously cooling a recycle stream of the hot mixture and then mixing the cooled recycle stream thoroughly with the main agitated body of the grease. We have found that greases may be produced in this manner having superior texture and other properties and in much higher yields than are obtainable by the methods of preparation employed heretofore, including such methods wherein the whole mass of the grease is shock cooled either statically or with agitation.

The grease preparation is carried out by heating a grease mixture comprising a soap and an oleaginous liquid in suitable grease forming proportions up to a temperature above the melting point of the soap, so as to form a homogenous liquid mixture of the soap and the oleaginous liquid, and then cooling this mixture down from a temperature above its gelation temperature until a grease consistency is obtained by maintaining a body of the mixture with agitation, while passing a recycle stream through a cooler and returning it to the main body of the grease to thereby cool the latter to a temperature below its gelation temperature at a controlled or critical rate. As applied to a lithium hydroxy fatty acid grease, the critical bulk cooling rate is above about 4° F. per minute. This critical rate of cooling of the bulk of the grease applies particularly from a bulk temperature of about 400–350° F. down to a temperature below about 320° F., preferably below 300° F. The recycle stream is cooled during its passage through the cooler to a temperature below the gelation temperature of the grease, and is then remixed with the main bulk of the grease which is above the gelation temperature during at least part of the operation.

In the drawings, Fig. 1 is a diagrammatic illustration of one form of apparatus suitable for making grease according to the method of this invention; and Fig. 2 is a chart showing the effect of cooling rate upon yield for a lithium 12-hydroxy strearate grease.

Referring in more detail to Fig. 1, numeral 1 represents a jacketed grease kettle equipped with stirrer 2 and adapted to be heated at elevated temperatures above about 400° F. A grease mixture, comprising a slurry of a soap and an oleaginous liquid, obtained either by introducing a preformed soap and an oleaginous liquid into the kettle or by saponification in situ, is heated in the kettle at an elevated temperature above the melting point of the soap for a sufficient time to obtain a completely homogeneous mixture of soap and oil. When the slurry is obtained by saponification in situ, a soap-forming fatty material is charged into the kettle together with all or a portion of the oleaginous material included in the grease and a solution of a suitable basic material introduced from tank 3 by way of line 4. The contents of the kettle are then heated with stirring until the saponification is complete and for a further period at a somewhat higher temperature to accomplish dehydration. When only a portion of the oleaginous liquid included in the grease is added to the original charge, the remainder may be suitably added following the dehydration. Any additives employed in the grease, such as oxidation inhibitors, corrosion inhibitors, etc., may be added suitably during the cooling down process, when the temperature of the grease is in the range between the melting point of the additive and its decomposition or volatilization temperature.

During the cooling down of the hot grease mixture, obtained as described above, a stream of the mixture is continuously circulated through heat exchanger 5, which may be any suitable heat exchanger which affords the necessary amount of cooling, such as a conventional shell and tube exchanger. The grease mixture passes to heat exchanger 5 through lines 6 and 8, containing valves 7 and 9, pump 10 and line 11, and is returned to kettle 1 by way of line 12. Circulation through heat exchanger 5 is begun while the grease is at a temperature above its gelation temperature, i. e., while it is still completely in a molten or homogeneous state, and is continued until a grease consistency is obtained. In some cases the circulation is advantageously continued for a considerably longer period, and if desired it may be continued until the grease is at a sufficiently low temperature for milling, or for packaging directly where milling is not employed. Stirring of the kettle contents is preferably employed during the cooling process in order to maintain a strong agitation of the grease mixture. The cooling of the grease mixture may be obtained substantially entirely by the cooling of the recycle stream, or additional cooling liquid through the kettle jacket. However, such additional cooling is ordinarily not required, and where cold water or other liquid is circulated through the kettle jacket, this should not be started until the grease structure has been established, in order to avoid graininess and loss in yield. Heating may also be applied to the kettle if desired in order to suitably control the rate of cooling of the grease.

When the grease is to be finished by milling, it is cooled in kettle 1, preferably to about 200° F. or lower, and then passed to mill 13, through lines 6 and 14, containing valves 7 and 15, pump 16 and line 17. From mill 13, the grease may pass by way of line 19 to kettle 18 for further cooling and for mixing with additional lubricating oil if desired in order to adjust the penetration and soap content. From kettle 18 the grease passes to packaging by way of line 20, valve 21, pump 22 and line 23.

The method of this invention is of particularly utility in the production of greases of the micro-crystalline type, such as lithium, aluminum and lead base greases, where the size of the soap crystals has a very critical effect upon both the lubricating characteristics of the greases and upon the yields obtained. Greases of this type are produced conventionally by shock cooling in order to obtain satisfactory smoothness. The production of greases thickened with lithium soaps of hydroxy fatty acids is regarded as a particularly important application of the invention, since such greases have heretofore required cooling in very thin layers, such as not more than about ¼ inch in thickness, in order to obtain both satisfactory texture and yield. By carrying out the preparation of these greases in the manner herein described, the commercially undesirable method of cooling in very thin layers is avoided and in addition very large reductions in the amount of the expensive lithium hydroxy fatty acid soap required for a given grease penetration have been obtained.

Suitable soap-forming hydroxy fatty acid materials which may be employed in the production of the lithium

hydroxy fatty acid greases are essentially saturated hydroxy fatty acids containing twelve or more carbon atoms and one or more hydroxyl radicals separated from the carboxyl group by at least one carbon atom, and the glycerides of such acids. Preferably the acid contains about sixteen to about twenty-two carbon atoms. Such materials may be obtained from naturally occurring glycerides, by hydroxylation of fatty acids, by hydrogenation of ricinoleic acid or castor oil, or otherwise by processes such as the catalytic oxidation of hydrocarbon oils and waxes which have been extracted and fractionated to the desired molecular range. Particularly suitable materials of this character are hydrogenated castor oil and 12-hydroxy stearic acid.

The oleaginous liquids employed in these greases may be any suitable oils of lubricating characteristics, including the conventional mineral lubricating oils, the synthetic lubricating oils prepared by cracking and polymerizing products of the Fischer-Tropsch process and the like, and synethtic oleaginous compounds within the lubricating oil viscosity range and mixtures thereof. The synthetic oleaginous compounds are those organic compounds which possess lubricating characteristics and may be substituted in whole or in part for the conventional mineral lubricating oils. Examples of these compounds are the aliphatic dicarboxylic acid di-esters, such as di-2-ethyl hexyl sebacate, di(secondary amyl) sebacate, di-2-ethyl hexyl azelate, di-isooctyl adipate, etc. Suitable mineral oils are those having viscosities in the range from about 100 to 2,000 seconds Saybolt Universal at 100° F., and may be either naphthenic or paraffinic in type, or blends of the two. When the saponification is carried out in the presence of a portion of the oil included in the grease, an oil which is not hydrolyzed under the saponification conditions is preferably employed for this purpose, most suitably a mineral oil fraction.

The greases may also contain various additives of the usual type such as corrosion inhibitors, oxidation inhibitors, antiwear agents, and so forth. Preferably, they contain an oxidation inhibitor, which may suitably be an oxidation inhibitor of the amine type, such as diphenylamine, phenyl alpha naphthylamine or tetramethyl diamino diphenyl methane. Compounds of this type may be added either before or during the cooling down process. They are preferably added while the temperature of the grease is between about 300° F. and about 180° F.

As an example of a preferred embodiment of the invention, a series of greases was prepared containing about 5–15% by weight of lithium 12-hydroxy stearate as the thickening agent and a base oil which was a blend of a refined paraffinic distillate oil and a steam reduced and propane deasphalted naphthenic residual oil in a 35:65 proportion by weight. Typical inspection tests on these oils were as follows:

	Distillate oil	Residual oil
Gravity, ° API	31.3	21.5
Flash, COC, ° F	410	550
Fire, COC, ° F	460	630
Viscosity, SUS, at 100° F	164.8	4,826
Viscosity, SUS, at 210° F		164.5
Pour, ° F	−10	45

In carrying out the preparation of these greases, a grease mixture was obtained by saponifying a commercial 12-hydroxy stearic acid with an approximately 10% aqueous solution of lithium hydroxide in the presence of the paraffinic distillate oil included in the grease. The saponification was carried out at a temperature of about 160–220° F. for about one hour and the mass dehydrated by heating for an additional hour at about 270–300° F. with stirring. Following the dehydration the residual oil was added, and the mixture then heated to about 380–425° F. and maintained at this temperature for about 30 minutes, until the soap was completely melted. The hot grease mixtures thus obtained were cooled both by conventional methods and by the method of this invention, and were finished by milling in a Premier colloid mill. When the temperature of the grease was at about 200° F. a small amount of diphenylamine, equal to 0.5% by weight of the finished grease, was added as an oxidation inhibitor. The greases were prepared in about 60–120 pound batches.

The above greases were cooled both by the method of this invention, by passing a recycle stream of the grease through an external cooler during the period while a grease structure was being formed, and also for comparison by the older methods of cooling in thin layers and by once through passage through a heat exchanger. In the grease preparations carried out according to this invention, the recycle stream was passed through either air cooled pipes or through a water cooled Graham Heliflow exchanger, consisting essentially of 9 helical ½ inch tubes having about 9 square feet of cooling surface surrounded by a water jacket, whereby the recycle stream at the cooler outlet, and before being remixed with the main bulk of the grease in the kettle, had a temperature below the gelation temperature thereof, generally below 320° F., such as about 260–300° F. The grease was recycled through these coolers at various rates from about 0.5 to about 30 gallons per minute, resulting in cooling rates of the bulk of the grease in the kettle from about 3.2° F. to about 100° F. per minute. The recycling was initiated while the temperature of the bulk grease mixture in the kettle was above about 350° F., generally about 375° F., and continued until the bulk grease had cooled to at least about 320° F., generally to below 300° F. Stirring of the kettle contents was employed during the recycling in order to provide additional agitation. Further cooling of the grease mixture down to about 200° F. was carried out by passing cooling water through the kettle jacket, while the stirring was continued. In some cases water was also passed through the kettle jacket during the recycling when the grease temperature was below 320° F. and after a grease structure had been established.

When the cooling was carried out as described above, the gelation process took place in a very different manner than when the cooling was carried out either statically or by once through passage through a heat exchanger, apparently due to the avoidance of supercooling by continually seeding the mass with fine soap crystals contained in the recycled stream. Thus, with recycle cooling, gelation of these grease mixtures began at temperatures up to about 375° F., or about 25° F. higher than with the other cooling methods. Also, very smooth greases were produced in good yields over the range of bulk cooling rates from about 4.7° F. per minute up to 100° F. per minute while the agitated bulk of the grease was being cooled in the 400–300° F. temperature range. However, when the cooling rate of the bulk of the grease by this recycle method was reduced below 4° F. per minute, such as 3.2° F. per minute, within the 400–300° F. range, the unexpected advantage in yield was not obtained, showing that a cooling rate of above 4° F. per minute is critical. With both static cooling and cooling by once through passage through a cooler, on the other hand, high cooling rates resulted in decreased yields; and with static cooling, cooling rates below about 25° F. per minute produced grainy and unsatisfactory greases. With the recycle procedure, cooling at higher rates above about 5° F. per minute within the 400–300° F. range had substantially no further improving effect upon the yields obtained. Below about 300° F. bulk temperature of the grease, the cooling rate by the recycle procedure had little or no effect upon the yield, when bulk cooling rates in the 300–200° F. range were varied from about 0.8° F.

per minute up to about 50° F. per minute. Consequently, it is seen that an important feature of the present invention is to cool the recycle stream below the gelation temperature thereof, and to return the cooled recycle stream to the agitated bulk of the grease so as to cool the latter from a temperature above its gelation temperature to a temperature below its gelation temperature at a cooling rate above about 4° F. per minute.

The greases produced by the recycle cooling method were smooth glossy greases, fully equivalent in appearance and texture to those obtained by cooling in ¼ inch layers, and superior in this respect to those obtained by cooling by once through passage through a heat exchanger. In addition, the yields obtained were much superior to those obtainable when the other methods of cooling were employed. By cooling with recycling at rates in excess of about 4° F. while the grease was in the 400–300° F. temperature range, NLGI No. 2 grade greases (265–295 ASTM worked penetration at 77° F.) were obtained consistently with soap contents of 5–7 percent of lithium 12-hydroxy stearate. This is a very striking and unexpected improvement over the yields obtainable with the other cooling methods. About 10 percent of lithium 12-hydroxy stearate is generally required to produce a grease of this grade by the static cooling method, and about 8–10 percent of this soap is required when the grease is cooled by once-through passage through a heat exchanger.

Fig. 2 shows the relationship between the cooling rate from 400° F. to 300° F. employed in the production of these lithium 12-hydroxy stearate greases and the yields obtained by the recycle cooling method of the present invention. As shown by the chart, cooling rates above about 4–5° F. per minute had little effect on the soap contents of the greases, resulting in products having 5.5% to 6.5% soap for a 280 penetration. However, the cooling rate becomes critical at a value of about 4° F., or between 4° F. and 5° F., per minute, as shown by the fact that the median curve shows a sharp rise of soap contents at this value.

Tables I, II, and III below show the results obtained in typical preparations carried out as described above in comparison with the results obtained by preparations employing the conventional methods of cooling. Table I gives typical results obtained by static cooling in thin layers. Table II gives results obtained by cooling the grease mixtures by once through passage through a heat exchanger. In these preparations the grease mixtures were heated to temperatures above 400° F. in a high temperature kettle and then cooled to temperatures below 300° F. by pumping them at different rates through a water cooled Graham Heliflow heat exchanger into a second kettle K–3, where they were further cooled at a slower rate with stirring. Table III gives results obtained by cooling the grease mixtures according to the method of this invention. The greases prepared by the recycle cooling which were made to initial soap contents of 7, 10 and 15% were too hard and had to be cut back to give greases of No. 2 grade as shown in the table. Mineral oil, comprising a 35:65 blend of the distillate and the residual oil, was added to these greases before the milling process. Greases No. 1 and 3 of Table I were obtained by statically cooling portions of the grease mixtures employed for greases No. 13 and 11, respectively, of Table III. The estimated soap content for a 280 penetration grease given in the tables was obtained by allowing 1% of soap for 20 points of difference in penetration.

TABLE I

Grease preparation with static cooling in thin layers

Grease No.	1	2	3	4
Soap content, percent	6.5	10	15	10
Soap content, estimated for 280 worked pene	9.5	10	13	9
Layer thickness, inches	¼	¼	¼	2
Cooling rate, ° F./min. (average values):				
400–300° F. range	25	25	25	2
300–200° F. range	15	15	15	1.7
Tests on milled product, penetration, ASTM:				
Unworked	350	268	260	241
Worked	340	275	238	247
Appearance	(1)	(1)	(1)	(2)

[1] Good.
[2] Grainy.

TABLE II

Grease preparation with cooling by once through passage through exchanger

Grease No.	5	6	7	8
Soap content, calc., percent	11	9	7.5	8
Soap content, estimated for 280 worked pene	10.55	10.7	8.6	8.1
Grease rate through cooler, gal./min.	4.6	10.2	1.9	2.9
Grease temperature, cooler outlet, ° F.	266	265	275	280
Cooling rate in cooler, ° F./min	31.4	85	18.3	22
Cooling rate in K–3, ° F./min	2.36	3.3	2.8	2.14
Tests on milled product:				
Free alkali (as LiOH), percent	0.17	0.12	0.16	
Free fatty acid (as oleic), percent	None	None		
Free neutral fat, percent	0.30	0.28	0.67	
Penetration, ASTM:				
Unworked	251	299	284	260
Worked, 60 strokes	271	314	302	282
Appearance	(1)	(1)	(1)	(2)

[1] Slightly hazy.
[2] Slightly grainy.

TABLE III

Grease preparation with recycle cooling

Grease No.	9	10	11	12	13	14
Soap content, percent:						
Original	7	10	15	7	6.5	5.5
Final	5.4	6.5	7.0	6.0	6.5	5.5
Soap content, estimated for 280 worked pene	5.55	6.35	7.0	5.55	5.75	5.75
Cooling rate, ° F./min.:						
400–300° F. range	7.2	4.7	4.7	35	100	17.1
300–200° F. range	2.7	2.2	2.3	4.0	9.7	2.75
Condition of recycling:						
Temperature started, ° F	387	350	389	395	400	400
Temperature stopped, ° F	232	290	320	200	220	202
Circulation rate, gal./min	29.92	29.92	29.92	28.13	28.13	1.05
Exchanger	(1)	(1)	(1)	(2)	(2)	(2)
Tests on milled product:						
Free alkali (as LiOH), percent	0.01	0.11	0.07		0.17	0.21
Free fatty acid (as oleic), percent	None	None				
Free neutral fat, percent	0.1	1.7	1.7		1.0	0.95
Penetration, ASTM:						
Unworked	285	283	285	245	265	285
Worked	283	277	280	271	269	285
Appearance	Good	Good	Good	Good	Good	Good

[1] Air cooled pipe.
[2] Water cooled Heliflow.

The data given in the above tables illustrate the large improvement in yield obtainable by carrying out the grease preparation according to the method of our invention. As shown by the data given in Table III, 280 penetration greases were obtained with as little as 5.55% of lithium 12-hydroxy stearate, whereas about 8–10% of this soap was required to produce a grease of equivalent grade when either pan cooling or cooling by once through passage through a heat exchanger was employed (Tables I and II). Also, as shown by the penetration data given in Table III, the greases thus produced were remarkably stable and resistant to change upon working, being superior both in stability and in appearance to the greases obtained by once through passage through a heat exchanger.

The following Table IV sets forth further data and results obtained by the recycle method of the present invention in the preparation of the lithium 12-hydroxy stearate greases of the type described above containing the distillate-residual mineral oil lubricating base:

TABLE IV

Grease No.	15	16	17
Circulation rate of recycle stream, gal./min.	0.59	0.47	14.1
Cooling rate of recycle stream, ° F./min.	17.7	18.9	140
Cooling rate, bulk of grease from 400° F. to 300° F., ° F./min.	4.7	3.2	33.3
Maximum temperature of recycle stream at cooler outlet, ° F.	280	266	294
Cooling rate, bulk of grease from 300° F. to 200° F., ° F./min.	0.8	1.1	[1] 15
ASTM worked penetration at 77° F. of grease for 6% soap content	270	331	277

[1] Cooled down to 245° F.

The foregoing run 15 in comparison with run 16 illustrates the criticality of the minimum bulk cooling rate in excess of about 4° F. per minute within the temperature range of 400–300° F. for the agitated bulk of the grease. Thus grease 15 with a soap content of 6% and made by the recycle procedure with a bulk cooling rate in the 400–300° F. range of 4.7° F. per minute, had a penetration of 270, which is on the hard side of the NLGI No. 2 grade. However, grease 16 with the same 6% soap content, but prepared by the recycle procedure with a bulk cooling rate in the 400–300° F. range of 3.2° F. per minute had a penetration of 331, indicating that approximately 8% soap would be required to bring this grease to the NLGI No. 2 grade. On the other hand, grease 17 prepared by the recycle procedure with a much higher bulk cooling rate of 33.3° F. per minute in the 400–300° F. range gave no further improvement in yield than grease 15, as shown by the penetration of 277 at 6% soap content. The actual rate of circulation of the recycle stream, or the cooling rate of the recycle stream, can vary widely, as illustrated by runs 15 and 17, and the desirable results of the present invention will still be obtained so long as the critical factors of bulk cooling rate in the 400–300° F. range above 4° F. per minute, and the maximum temperature of the recycle stream at the cooler outlet below the gelation temperature, are maintained. Also, the bulk cooling rate below 300° F. can also vary widely without deleterious results so long as the above noted critical factors are maintained, as again shown by runs 15 and 17.

Obviously many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof and, therefore, only such limitations should be imposed as are indicated in the appended claims.

We claim:

1. The method of grease manufacture which comprises providing a molten homogeneous mixture of a lithium hydroxy fatty acid soap and an oleaginous liquid in grease forming proportions at an elevated temperature above the melting point of the soap, and controlling the crystallization of the said soap during cooling by maintaining a body of relatively large volume of the said mixture with agitation while reducing the temperature thereof until a grease consistency is obtained by continuously withdrawing a stream of relatively small volume from said body of mixture, passing the said stream through an external cooler where it is cooled to a temperature below the gelation temperature of the said mixture and returning the said cooled stream to the maintained body of mixture.

2. The method of preparing a grease containing as the thickening agent a lithium soap of a soap-forming hydroxy fatty acid material, which comprises providing a molten homogeneous mixture of said lithium soap and an oleaginous liquid in grease forming proportions at a temperature above about 350° F., and controlling the crystallization of the said soap during cooling by maintaining a body of relatively large volume of the said mixture with agitation while reducing the temperature thereof from above about 350° F. down to at least about 320° F. by continuously withdrawing a stream of relatively small volume from said body of mixture, passing the said stream through an external cooler where it is cooled to a temperature below about 350° F., and returning the said cooled stream to the maintained body of mixture.

3. The method of preparing a grease containing as the thickening agent a lithium soap of a soap-forming hydroxy fatty acid material which comprises providing a molten homogeneous mixture of said lithium soap and an oleaginous liquid in grease forming proportions at a temperature above about 375° F., and controlling the crystallization of the said soap during cooling by maintaining a body of relatively large volume of the said mixture with agitation while reducing the temperature down to about 300° F. at an average rate above about 4° F. per minute by continuously withdrawing a stream of relatively small volume from said body of mixture, passing the said stream through an external cooler where it is cooled to a temperature below about 350° F. and below the gelation temperature thereof, and returning the said cooled stream to the maintained body of mixture.

4. Claim 3 wherein the said grease is finished by milling at a temperature below about 200° F.

5. The method of preparing a grease contaning as the thickening agent a lithium soap of a soap-forming hydroxy fatty acid material which comprises providing a molten homogeneous mixture of said lithium soap and an oleaginous liquid in grease forming proportions at a temperature of about 400° F., and controlling the crystallization of the said soap during cooling by maintaining a body of relatively large volume of the said mixture while reducing the temperature from about 400° F. to about 300° F. with agitation at an average rate of about 4–100° F. per minute by continuously withdrawing a stream of relatively small volume from said body of mixture, passing the said stream through an external cooler where the temperature is reduced below the gelation temperature of the recycle stream, and returning the said cooled stream to the maintained body of mixture.

6. The method of preparing a grease containing as the thickening agent a lithium soap of a soap-forming hydroxy fatty acid material which comprises forming the lithium soap by saponification in the presence of mineral oil comprising a portion of the total oleaginous liquid included in the grease, dehydrating, adding the remainder of the oleaginous liquid, heating the mixture of soap and oleaginous liquid thus obtained to about 380–425° F. to obtain a molten homogeneous body of said mixture, cooling to about 200° F. and milling, the crystallization of the said soap being controlled during the cooling by maintaining a body of relatively large volume of the said mixture with agitation while reducing the temperature from about 400° F. to about 300° F. at an average

rate of about 4–100° F. per minute by continuously withdrawing a stream of relatively small volume from said body of mixture, passing the said stream through an external cooler where it is cooled down to a temperature below about 300° F., and returning the said cooled stream to the maintained body of mixture.

7. The method of grease manufacture which comprises providing a molten homogeneous mixture of a lithium hydroxy fatty acid soap and an oleaginous liquid in grease forming proportions at an elevated temperature above the melting point of the soap, and controlling the crystallization of the said soap during cooling by maintaining a body of relatively large volume of the said mixture with agitation while reducing the temperature thereof until a grease consistency is obtained by continuously withdrawing a stream of relatively small volume from said body of mixture, passing the said stream through an external cooler where it is cooled to a temperature below the gelation temperature of the said mixture at a rate of at least about 17° F. per minute, and returning the said cooled stream to the maintained body of mixture to provide a bulk cooling rate of the maintaining body of above 4° F. per minute.

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