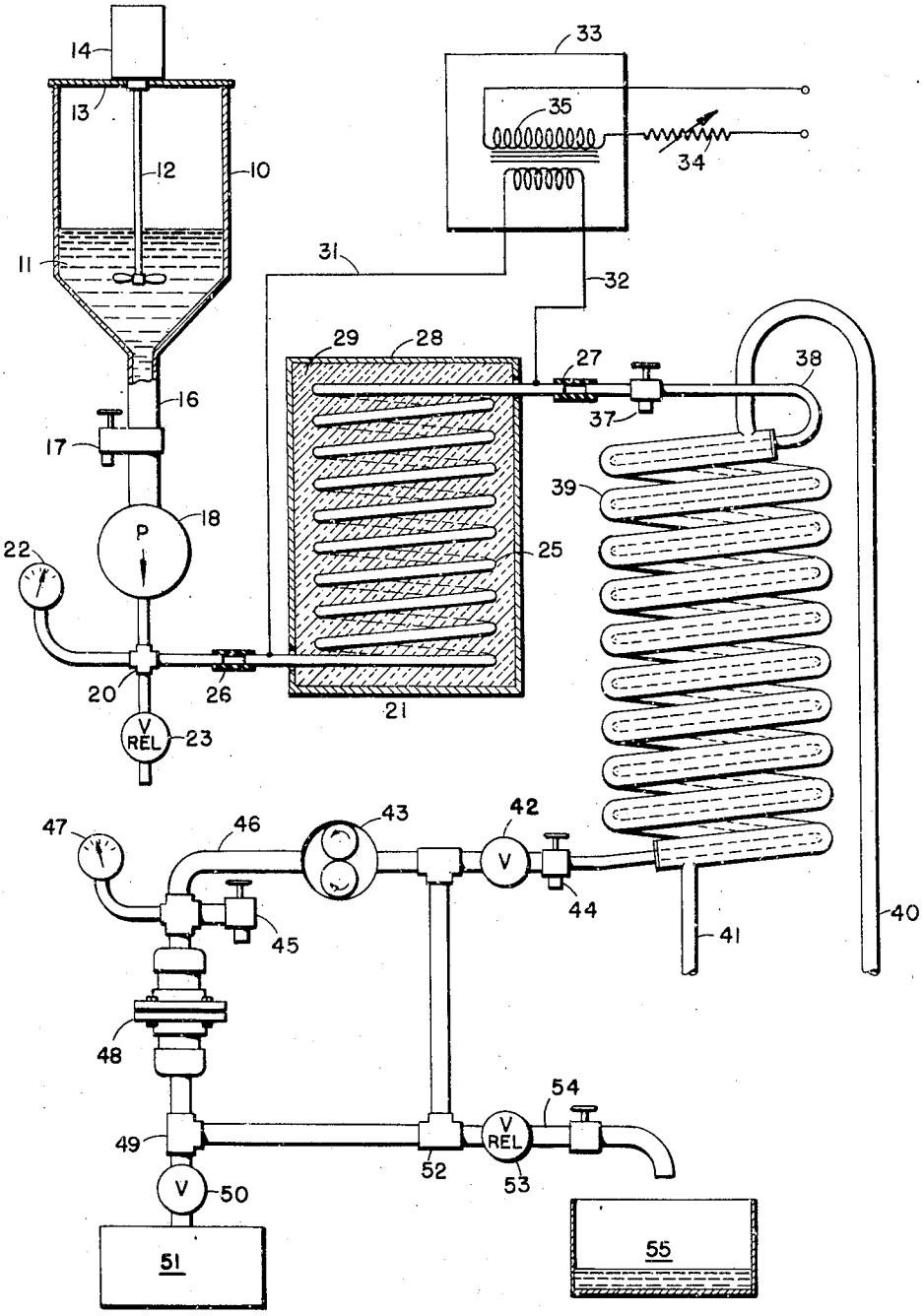
Aug. 16, 1949. G. M. HAIN 2,478,917

METHOD AND APPARATUS FOR MAKING GREASE

Filed Sept. 24, 1946 3 Sheets-Sheet 1



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METHOD AND APPARATUS FOR MAKING GREASE

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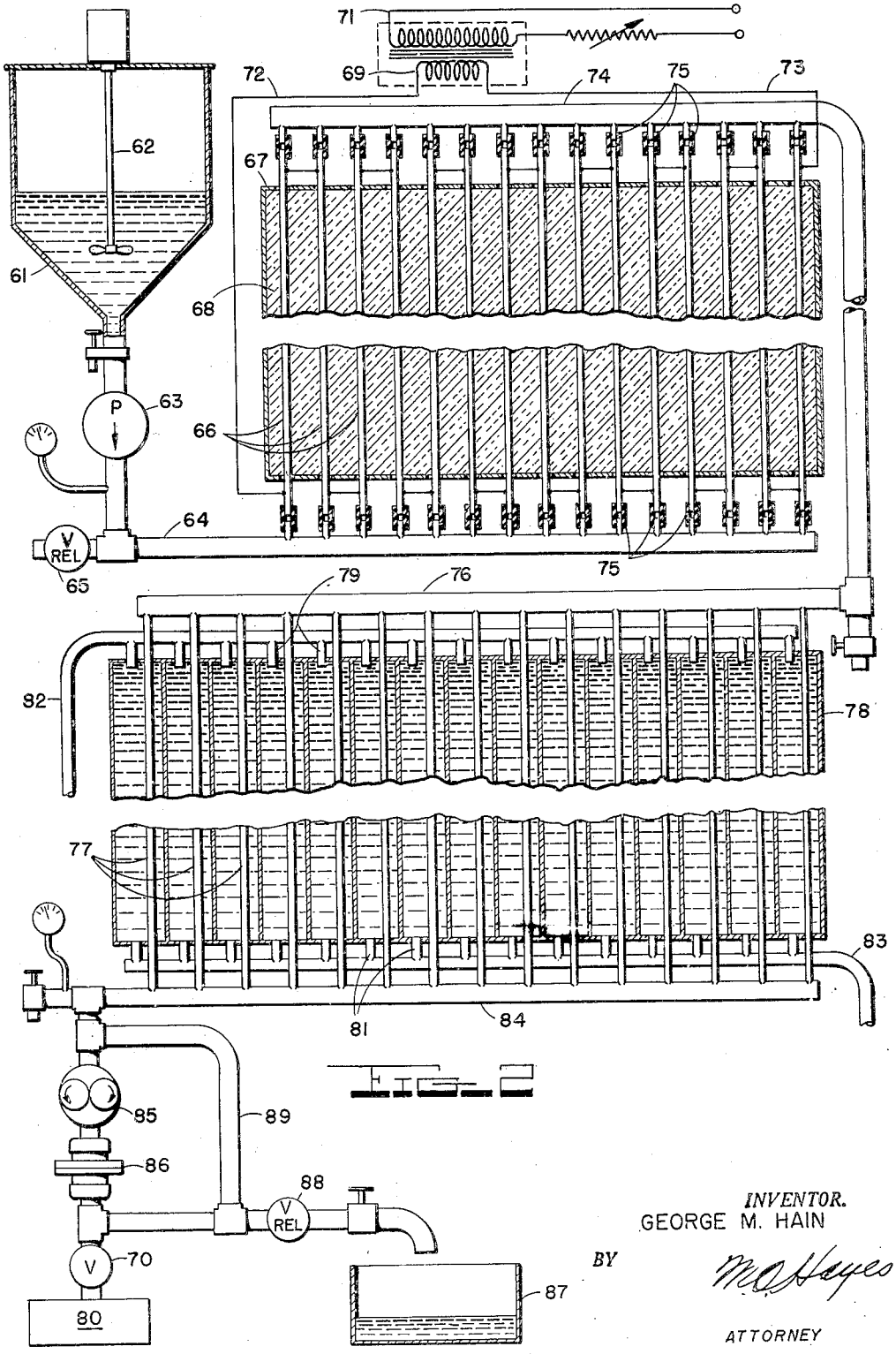


FIG. 2

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METHOD AND APPARATUS FOR MAKING GREASE

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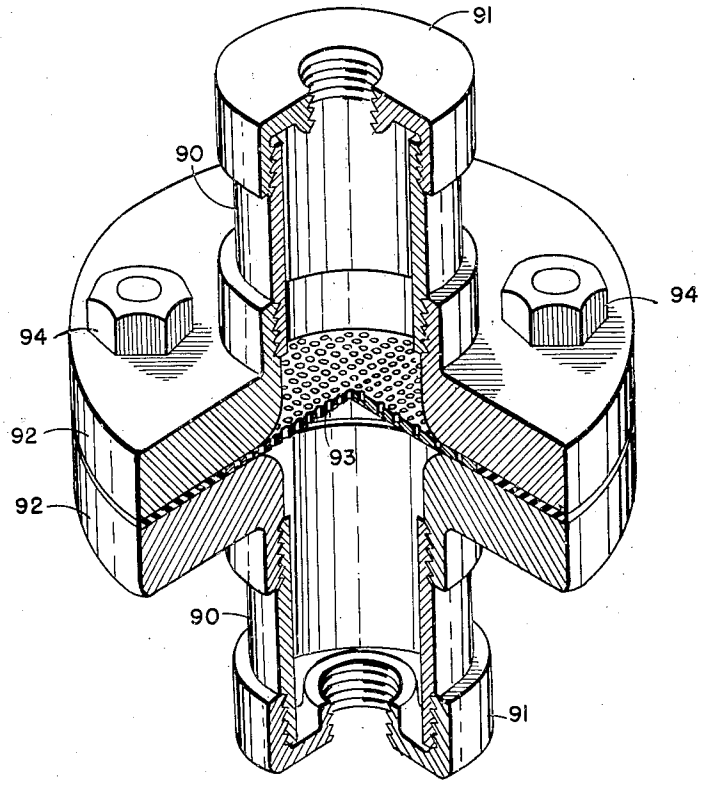


FIG. 3

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METHOD AND APPARATUS FOR MAKING GREASE

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6 Claims. (Cl. 252—32)

(Granted under the act of March 3, 1883, as amended April 30, 1928; 370 O. G. 757)

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This invention relates to a method and apparatus for the manufacture of high quality greases by a continuous flow process.

The art of manufacturing high quality greases is recognized as a highly specialized phase of lubrication chemistry and is fraught with numerous difficulties. The control of sensitive variables must be closely maintained in order that operating conditions can be changed rapidly and accurately.

Continuous processes for grease manufacture generally include the successive steps of heating an intimately mixed slurry of prepared soap in a mineral oil or other base fluid medium to achieve complete solution of the soap therein, the sudden cooling of the solution thus obtained to form a gel, and the mechanical working of the gel to a grease consistency. Heretofore, both the apparatus and methods employed in such processes have in general proven unsatisfactory when applied to quality grease manufacture.

High-quality greases are critical compositions comprising essentially loosely flocculated dispersions of tiny soap crystals in a fluid medium, the size and state of aggregation of the crystals having a pronounced effect on the physical properties of the finished grease. A microcrystalline soap structure of large inter-crystalline surface area is necessary in such a grease to hold the fluid phase in equilibrium and prevent its bleeding from the mass. However, none of the known continuous or semi-continuous processes for making greases are operative to produce the optimum microcrystalline dispersion of soap.

In the preparation of many greases having the desired microcrystalline soap structure, the transition from hot fluid to stable gel must be accomplished as quickly as possible, since slow cooling promotes crystal growth and therefore less interfacial area to hold the fluid. As an aid to the desirable rapid transition, the heating step should not be carried to a temperature materially beyond that needed to complete solution of the soap in the base fluid. Further, the cooling should proceed to equilibrium conditions at such a rate as to avoid the formation of coarse soap granules.

Accordingly, it is an object of my invention to provide a method and apparatus for the manufacture of high quality greases, such as lithium soap greases, by a continuous process which affords complete control of operating conditions at all stages.

Another object of my invention is to provide

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a method and apparatus for the continuous manufacture of greases which method and apparatus create a condition whereby the grease is subjected to internal working in several successive stages.

It is another object of my invention to provide a continuous flow method and apparatus by which it is possible to convert a fluid grease formulation into a stable grease characterized by its stability and homogeneity.

Another object of my invention is to provide a simple apparatus for the continuous manufacture of high quality greases, which apparatus is characterized by its having a restricted cross section in its heating and cooling sections.

It is another object of my invention to provide an apparatus having heating and cooling sections wherein essentially film type heat transfer occurs and heating and cooling of the composition occurs at very rapid rates.

Further and more specific objects will in part be obvious and in part appear hereinafter.

My invention, accordingly, comprises the process of making high quality greases in continuous flow manner, which process includes the several steps and the relationship of one or more of such steps to each of the others, and the apparatus comprising successive mixing, heating, cooling, and working sections which possess the features of construction and combinations of elements exemplified in the following detailed disclosure. The scope of the invention will be indicated in the appended claims.

The process is carried out by preparing a fluid grease formulation which consists of the fluid base for the grease having suspended therein the soap thickener, a typical one of which is lithium stearate, and various balancing ingredients such as oiliness, oxidation inhibiting, rust inhibiting additives and the like as may be required in the final grease formulation. The fluid formulation is conducted through successive heating and cooling steps in a continuous flow method in essentially film form, so that the heating and cooling are characterized by being carried out as film heating and cooling heat transfer operations. Rather than heating and cooling a single large mass of fluid in a vat or conduit, a continuously flowing fluid having a cross section of the order of the thickness of a flowing film of the base fluid is subjected to the heating and cooling operations.

The apparatus in which the process is carried out is characterized by structure which maintains the flowing, fluid, grease formulation in the

form of a fast moving film. It is further characterized by being made up of small diameter thin-walled conduits in which the largest diameter is of the order of twice the thickness of a free flowing film of the base fluid. By employing such restricted diameter throughout, and maintaining a high velocity of flow of the film through the conduits, a precise control is maintained at all times over the temperature of the fluid, and in addition, the fluid, particularly in the cooling stage where it is converted to a gel, is subjected to internal shearing stresses which impart to it a certain amount of work during the thermal treatment. Additionally, the velocity of flow of the fluid through the heating and cooling sections is matched to the heating capacity and the cooling capacity of the apparatus so that at the exit from either section the composition is closely within a few degrees of the optimum temperature for that section.

The details of construction of the apparatus and the method of operation will be apparent from a consideration of the following detailed disclosure when taken in conjunction with the accompanying drawings wherein:

Figure 1 is a schematic diagram of an apparatus embodying the invention;

Figure 2 is a schematic diagram of a second embodiment of the invention;

Figure 3 is a perspective view, partially in section, showing the structure of the grease working section.

In Figure 1 there is shown diagrammatically a hopper 10 containing a soap-fluid base slurry 11, which slurry is maintained in a free-flowing state by a suitable mechanical stirring or mixing means 12 held in the hopper 10 by means of the support 13 and driven by motor 14. The slurry is conducted through pipe 16 which is fitted at its lower end with a drainage cock 17 to high pressure pump 18. The pump discharges slurry at a pressure of from about 500 to about 1,000 or 1,500 pounds per square inch through the junction 20 into the heating unit 21. The heating tube 25 of this unit is made of tubing preferably of stainless steel or other corrosion resistant metal, having an inside diameter of the order of about twice the thickness of a free-flowing film of the fluid base of the grease composition and a wall thickness of a few millimeters, that is, merely sufficient to hold the pressure developed. The significance of the tube diameter and the method of arriving at the specific dimensions will be outlined hereinafter. At the junction 20 are a pressure gage 22 and pressure-release safety valve 23.

The heating tube 25, electrically isolated from the rest of the apparatus by insulators 26 and 27, is enclosed in a container 28 in heat insulating material 29, such as glass wool. Electrical leads 31 and 32 serve to connect the heating coil across the secondary terminals of a transformer 33, which transformer has a current regulating device 34 inserted in series with its primary windings 35. At junction 36 there is connected a sampling tube 37 and a lead to the cooling coil 38. In order to maintain a condition of restricted cross section throughout the apparatus to subject the composition to continuous working, the tubing in the cooling coils is of the same cross section as that in the heating section thereby keeping the course of fluid flow in the apparatus small, that is of the order of twice the thickness of free-flowing film.

A water jacket 39 is provided for the cooling coil 38, water being introduced through the inlet 40, allowed to pass over and around the coil and leave through the outlet 41. This discharge end of the cooling section is connected by a lead through a valve 42 to a variable speed gear pump 43. Near this valve junction and also at the discharge end of the pump 43 are connected other sampling tubes 44 and 45. A pressure gage 47 is also connected at the discharge end of this pump which end is joined to the working section 48, the construction of which will be fully described hereinafter in connection with Figure 3. One end of a T junction 49 is connected to the discharge side of the working section, the other two ends being connected respectively through a valve 50 to a vacuumizing system 51 and through a pipe T 52 to the intake of the gear pump 43. As a discharge point for the apparatus a check valve 53 controls the flow of finished grease into the take-off tube 54 to the container 55.

In Figure 2 there is shown a second embodiment of my apparatus which can be employed for the production of greases on a large scale and the apparatus therein shown consists essentially of a plurality of units such as those described in Figure 1 arranged in parallel in order to multiply the capacity of grease production and yet maintain the forced-film-flow and heating and cooling conditions characteristic of the unit described in connection with Figure 1.

Figure 2 shows a slurry tank 61 with its associated mixing and pumping means 62 and 63. Cold slurry is discharged from the pump 63 into a header 64 which is fitted with a pressure release safety device 65. This header 64 feeds slurry into a plurality of narrow diameter, thin-walled heating tubes 66 which have dimensions equivalent to those of the heating tube described in connection with Figure 1. Throughout the major portions of their lengths, the heating tubes 66 are enclosed in a container 67 of heat insulating material 68. The tubes are electrically connected in series across the secondary windings 69 of a power transformer 71 by leads 72 and 73. Further, they are electrically isolated from other components of the apparatus by insulators 75 placed at each of their respective ends.

Hot grease solution is discharged under pressure from the heating tubes into a header 74, through which it is conducted to another header 76 which feeds a plurality of cooling tubes 77 having the same dimensions as the heating tubes. Each of the cooling tubes 77 is encased in an individual water-jacket 78 which is fitted with a water inlet 79 and a water outlet 81. The water inlets 79 are fed from a conduit 82. Water outlets 81 discharge into the other conduit 83 of the water supply system. The grease composition discharged from the cooling tubes 77 into the header 84 is picked up by a pump 85 and fed to the working device 86. Fixed minor portions of the finished grease composition may be taken off constantly to the receptacles 87 through a preset ball-check valve 88, while the larger portions of grease are recycled through the plumbing 89 to the intake of the pump 85 for reworking. Before the apparatus is placed in operation, it may be evacuated through a valve 70 which connects to an evacuating pump 80.

Referring to Figure 3, the working means comprises two symmetrical sections each consisting of a short length of large diameter pipe 90 having at one end, a pipe cap 91 which has a threaded hole through its center, and at the other end a pipe flange 92. A thin metal plate 93 having a

plurality of small perforations over a central portion of a diameter matching the inlet is sandwiched between the flat surfaces of the flanges, gasketed, and secured firmly by the bolts **94** which pass through the plate and flanges and join the top and bottom sections. The fluid conduits of the apparatus may be used to connect the working means with the pump **43** shown in Figure 1.

With reference to Figure 1, in preparation for starting, the system is evacuated by means of any convenient pumping equipment **51**. When a substantial reduction of the air pressure in the system has been accomplished, valve **50** is closed and the cold slurry of soap and fluid, which has been maintained in a free-flowing state by means of the mechanical stirring apparatus, is pumped at a pressure of about 800 pounds per square inch into the narrow diameter tubing of the heating coil. Here, the accurate control afforded by the unique form of heating apparatus employed permits attainment of complete solution of the soap crystals in the base fluid medium just as the fluid is discharged from the heating coil. This hot solution enters the cooling coil under pressure where it is transformed quickly into a semi-rigid gel, which gel in its forced passage through the cooling coil is worked at least partially to a grease consistency by the severe shearing stresses set up in the narrow diameter tubing comprising the coil.

The rapid succession of heating and cooling steps occurring in the heating and cooling sections of the apparatus minimizes fire hazards and the possibility of oxidation of the hot fluid base and also avoids damage to the soap particles and additives in the composition. The rate of flow through the narrow diameter heating section is adjusted so that the total time of heating does not materially exceed about 15 seconds when a fluid is brought from an ambient temperature of about 70° F. to a maximum temperature of discharge from the heating section of about 480° or 500° F. The exact velocity of flow through the heating and cooling sections therefore will be fixed by the dimensions of the apparatus and the specific physical properties of the base fluid which fix those dimensions but will roughly approximate about 50 cubic centimeters per minute in a tube having a diameter of about one-quarter of an inch.

The cool, partially worked grease composition is picked up by the gear pump **43** shown in Figure 1 and pumped into the working section where it is worked to the consistency of a finished grease by being extruded through the finely perforated plate. The grease first passing the working section is recycled into the gear pump until the plumbing associated with this pump and the working means has been filled with grease and a head of grease has been built up sufficient to activate the preset ball back valve **53**. Then, as grease continues to be forced out of the working means, which acts also as a reservoir for grease, a fixed minor portion is constantly taken off through the ball check valve as the finished product. The remaining grease is recycled into the pump and working section. By this method of constantly taking off only a small portion of finished grease and recycling the remaining portion, it will be apparent that each individual portion of finished grease has had at least several passes through the perforated plate of the working section.

During the process of manufacture, the occlusion of air to form detrimental pockets in the finished grease is substantially entirely prevented because of the high pressures employed, and the extensive working to which the grease is subjected.

Instead of employing the more or less conventional steam or water-jacketed pipe-still heating coils set in a furnace heated by coal, gas or oil fires, metal tubing, and preferably corrosion resistant stainless steel tubing, of very small diameter and wall thickness is employed and heated by an electric current. As seen from Figure 1, the tubing is electrically connected across the secondary terminals of the power transformer **33**. The current flowing through the tubing when the primary circuit of the transformer is energized produces heat at a steady even rate over the entire length of the coil. The rheostat **34** inserted in series with the primary windings of the transformer governs the amount of current flowing there and hence, by normal transformer action, governs the amount of current induced in the secondary windings and flowing through the heating section. The rheostat thus serves as an effective and accurate temperature control of the heating means. Because the heat is generated directly and evenly in the tubing walls, and because the tubing is of such narrow diameter, substantially instantaneous heat transfer is obtained. Furthermore, local overheating or variations in rate of heating, both of which are detrimental to the attainment of good grease structure, can be avoided. Since any given slurry is flowing through the heating section at a rate fixed by the pumping means employed, it is practicable to regulate the heating so that the temperature needed for complete solution of the soap in the fluid medium may be reached close to the discharge end of the coil and not be exceeded to any appreciable degree. As stated above, this temperature may for certain soaps be as high as from about 480° F. to about 500° F.; but in general it will be slightly above 300° F.

Substantially instantaneous heat exchange is also afforded in the cooling section by virtue of the employment of narrow diameter thin-wall tubing in its construction. It will be recognized by those skilled in the art that this point of control is very important with regard to the quality of grease produced. The solution must be cooled so rapidly that the fine soap crystals which precipitate and form the basis of a gel structure do not have time before equilibrium is reached to grow or agglomerate into the relatively large soap granules which permit the final grease composition to bleed. Furthermore, the solution must be cooled at the same rate throughout in order to produce a gel structure which is homogeneous throughout.

My unique cooling means affords the operational control necessary to the accomplishment of these ends. In addition, it provides a means of at least partially working the semi-rigid gel to a proper grease consistency. As the gel is formed in the narrow diameter cooling section, it is forced rapidly through the tubing and tangentially against the inner walls thereof at high rates of shear. The gel structure is partially broken down to produce a composition which on leaving the cooling section has a grease texture. More extensive working of the grease takes place in the working section of the apparatus.

Because of the high operating pressures and the narrow diameter system employed in this continuous flow method of grease manufacture I have attained what is essentially a self working system which develops high rates of shear substantially throughout. In the working section the shearing stresses are magnified greatly as the grease com-

position is forced through the tiny holes of the perforated plate.

In describing the heating and cooling sections of the apparatus, reference was made to maintaining a narrow diameter throughout these two sections preferably in the form of a narrow diameter tube which created a condition of flow in the apparatus which I chose to call a forced-film-flow. The exact dimensions of the tube, that is, the tube diameter, are not critical but should be of the order of about two times the thickness of the free-flowing film of the base fluid and for most grease fluids this is less than one-half or three-eighths of an inch.

To simplify a description of my apparatus and the operation thereof I have hitherto spoken of a slurry as comprising merely a mixture of soap crystals in a base fluid medium. It is to be understood that in addition to the soap and base fluid constituents there may be present in the slurry any of the common balancing ingredients such as oxidation and corrosion inhibitors, oiliness and extreme pressure additives, and polymer thickeners.

The particular soap and other additives are, in general, blended with the base fluid prior to its introduction into the apparatus. In other words, it is preferred that the compounding of the ingredients, including any necessary chemical reactions, occurs outside the apparatus.

Using the apparatus described, and following the methods described, instrument greases of high quality having various types of base fluids have been prepared by my continuous process at a rate of about 120 pounds per 8 hour day. The greases were all characterized by their being smooth homogeneous mixtures of a base fluid lubricant with a metallic soap and, when appropriate, various balancing ingredients. Various diesters and other synthetic fluids such as the silicones, hydrocarbon fluids, and polyethylene oxide fluids were used as base fluids. The dispersions of the soap in the grease in all cases were homogeneous and no objectionable base fluid separations occured even after months of standing in many cases. Typical formulations which were prepared by the above methods and in the above described apparatus are shown below in the table.

Table

No.	Components	Parts by Wgt.	A.S.T.M. Penetration	Wgt. Per Cent Bleeding	Wgt. Per Cent Evaporation
1	lithium stearate	12	245	1.90	0.00
	di-2-ethylhexyl sebacate	88			
2	lithium stearate	12.0	245	2.65	0.33
	polybutene (MW-12000)	1.0			
	4-tertiary-butyl-2-phenyl-phenol	0.2			
	di-2-ethylhexyl sebacate	34.8			
	di-2-ethylhexyl adipate	52.0			
3	lithium stearate	14.0	260	3.90	0.98
	4-tertiary-butyl-2-phenyl-phenol	0.2			
	sorbitan monooleate	2.0			
	polybutene (MW-12000)	1.0			
	di-2-ethylhexyl sebacate	82.8			

My invention thus provides a unique method and apparatus for the continuous manufacture of high quality greases, which method is characterized by its being a continuous process of forced-film-flow which works the grease composition in all stages of its manufacture. The apparatus is correspondingly a narrow diameter, self-working, self-scraping system in which high rates of shear within the composition are obtained. It affords close control of operational variables, and reduces the handling of raw materials thereby introducing a desirable factor of safety. Because it is essentially a closed system, my apparatus may be used to advantage in the manufacture of greases having toxic ingredients. Aeration of the grease produced with the resultant decrease in quality and increased tendency to bleed is prevented by using high working pressures throughout the system.

It will be apparent that many modifications may be made in my invention without departing from the scope thereof. Accordingly, it is desired that the present disclosure be construed as illustrative of my invention and not by way of limitation.

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

What is claimed is:

1. Apparatus for the continuous preparation of grease comprising a slurry mixing tank, a discharge conduit from said tank, a high pressure pump connected to said discharge conduit, a grease heating conduit connected to and electrically insulated from said high pressure pump, an electrical heating circuit connected through said grease heating conduit, a heat insulating medium surrounding said grease heating conduit, a grease cooling conduit connected to and electrically insulated from said grease heating conduit, a cooling medium conducting jacket enveloping said grease cooling conduit, said heating and said cooling conduits each being of a diameter substantially twice the thickness of a free-flowing film of the base fluid of the slurry, a second high pressure pump connected to the discharge side of said grease cooling conduit, a grease working section connected to the discharge side of said second high pressure pump, said grease working section including a finely perforated membrane through which the grease is forced, and check valved grease discharge means connected to said grease working section.

2. Apparatus for the continuous preparation of grease comprising a slurry mixing tank, a discharge conduit from said tank, a high pressure pump connected to said discharge conduit, a grease heating conduit connected to and electrically insulated from said high pressure pump, an electrical heating circuit connected through said grease heating conduit, a heat insulating medium surrounding said grease heating conduit, a grease cooling conduit connected to and electrically insulated from said grease heating conduit, a cooling medium conducting jacket enveloping said grease cooling conduit, said heating and said cooling conduits each being of a

diameter substantially twice the thickness of a free-flowing film of the base fluid of the slurry, a second high pressure pump connected to the discharge side of said grease cooling conduit, a grease working section connected to the discharge side of said second high pressure pump, said grease working section including a finely perforated membrane through which the grease is forced, a recycling connection between the entrance and discharge sides of said grease work section, and check valved grease discharge means connected to said grease working section.

3. Apparatus for the continuous preparation of grease comprising a slurry mixing tank, a discharge conduit from said tank, a high pressure pump connected to said discharge conduit, a header connected to said high pressure pump, a second header, grease heating means connected between and electrically insulated from said headers, said grease heating means comprising a plurality of individual grease heating conduits and an insulating medium through which said grease heating conduits extend, an electrical heating circuit connected through each of said heating conduits in series, a conduit from said second header to a third header, a plurality of grease cooling conduits connecting said third header to a fourth header, individual cooling medium conducting jackets enveloping each of said grease cooling conduits, said heating and said cooling conduits each being of a diameter substantially twice the thickness of a free-flowing film of the base fluid of the slurry, a second high pressure pump connected to the discharge side of said fourth header, a grease working section connected to the discharge side of said second high pressure pump, said grease working section including a finely perforated medium through which the grease is forced, and check valved grease discharge means connected to said grease working section.

4. Apparatus for continuous preparation of grease comprising a slurry mixing tank, a discharge conduit from said tank, a high pressure pump connected to said discharge conduit, a header connected to said high pressure pump, a second header, grease heating means connected between and electrically insulated from said headers, said grease heating means comprising a plurality of individual grease heating conduits and an insulating medium through which said grease heating conduits extend, an electrical heating circuit connected through each of said heating conduits in series, a conduit from said second header to a third header, a plurality of grease cooling conduits connecting said third header to a fourth header, individual cooling

medium conducting jackets enveloping each of said grease cooling conduits, said heating and said cooling conduits each being of a diameter substantially twice the thickness of a free-flowing film of the base fluid of the slurry, a second high pressure pump connected to the discharge side of said fourth header, a grease working section connected to the discharge side of said second high pressure pump, said grease working section including a finely perforated membrane through which the grease is forced, a recycling connection between the entrance and discharge sides of said grease work section, and check valved grease discharge means connected to said grease working section.

5. Apparatus for the continuous preparation of grease, comprising, a slurry mixing tank provided with a discharge conduit, a high pressure pump connected with said discharge conduit, a heating conduit connected to and electrically insulated from said high pressure pump, means for passing electric current through said heating conduit, a cooling conduit provided with a cooling medium conducting jacket and connected to said heating conduit and electrically insulated therefrom, said heating and cooling conduits being of a diameter not greater than approximately one-half inch, a second high pressure pump connected to the discharge side of said grease cooling conduit, and a grease working section including a finely perforated membrane through which the grease is forced connected to the discharge side of said second pump.

6. Apparatus of the type defined in claim 5 wherein the worker section comprises two substantially symmetrical axially apertured flange members axially aligned with each other, a thin perforated metal plate positioned between the faces of said member in gasketed relation therewith and extending across said axial apertures, and bolt means holding said flange members in rigid, face-to-face relationship.

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