

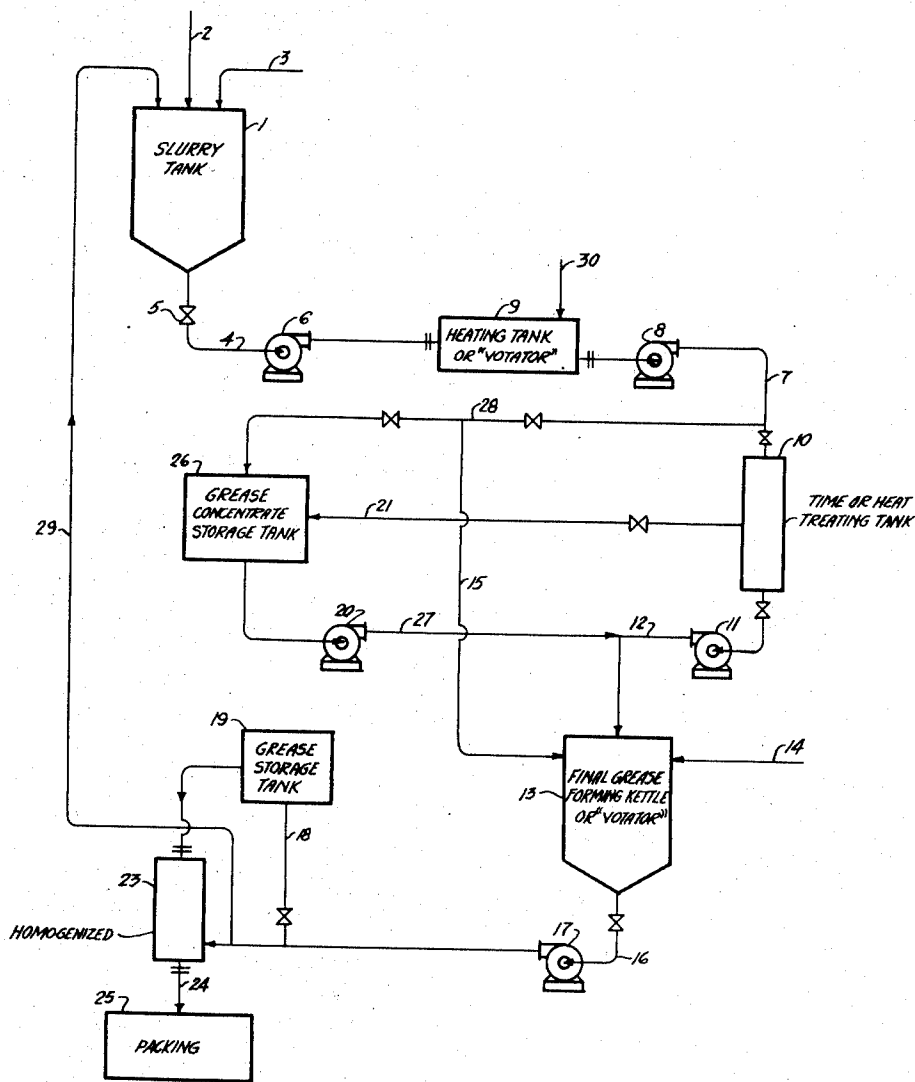
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MANUFACTURE OF GREASE COMPOSITIONS

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MANUFACTURE OF GREASE COMPOSITIONS

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This invention relates to a new and improved method of making grease compositions. More particularly this invention pertains to an improved method of making wide temperature lubricating grease compositions which are extremely stable under static and dynamic conditions.

It is well established that the quality of a grease varies substantially with the method of making it. Thus lubricating grease compositions made from identical materials but in which the method of making said grease is varied will in one case result in a grease of excellent quality and in another case be extremely poor.

One of the most important and controlling factors in the manufacture of a good grease is the selection of a proper base oil for the soap or gelling agent used in making the grease. For example, in the manufacture of lithium soap greases suitable for low temperature use a light viscosity base oil must be used. However, such oils present a serious problem in that they present a fire hazard problem since in making grease an elevated temperature must be used. Also greases made from light oils are at times incapable of retaining the grease structure, particularly under dynamic conditions, resulting in poor lubrication of bearing surfaces and bleeding under storage or stable conditions. The same problem is also presented with soaps other than lithium soaps. Very high viscosity oils are also to be avoided for making greases for it is difficult to disperse soaps in such oils inasmuch as soap fibers which ultimately govern the quality of a grease have little if any change of growing to a fibrous or micelle structure in such oils so as to form a grease of good stability.

It is an object of this invention to produce greases by a novel process. Another object of this invention is to produce greases by a novel process which are stable under static and dynamic conditions. Another object of this invention is to produce greases by a novel process which resists bleeding. Still another object of this invention is to provide a method of making greases having a definite micelle or soap structure. Still another object of this invention is to provide a method of making greases in a batch or continuous process whereby mechanically stable, non-bleeding products are produced. Still another object of this invention is to produce a grease of desired consistency with a minimum amount of soap.

Broadly, this invention comprises a method of making improved stable greases involving two essential features: (1) the making of a grease

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concentrate containing from 10% to 50% gelling agent, e. g. soaps, said concentrate being made in any conventional way using a highly viscous oil, the viscosity of which must be above 500 at 100° F. SUS and (2) diluting said concentrate at any convenient time with a light oil having a viscosity of less than 300 at 100° F. SUS, such as a spray oil, the amount of light oil used being sufficient so that the final soap content based on the total composition varies from 4% to 15% by weight.

A particularly novel feature of this invention is that the grease concentrate when prepared with a viscous oil as outlined above may be diluted in the cold or heated only slightly to say 80° or 90° C. prior to or while the light oil is being incorporated so as to form the final grease product. This feature is not only novel from the point of view that by the process of this invention grease compositions can be made at low temperatures resulting in a marked saving in fuel, but results in a saving in the handling and transporting of large quantities of material and eliminates the use of expensive equipment necessary for making greases. Thus, for example, a grease concentrate of this invention can be prepared, packaged and shipped to a desired destination, where it is simply admixed either at room or slightly elevated temperature with the required amount of a suitable light oil and is ready for use.

As can be noted the essence of this invention is to prepare a grease concentrate using a highly viscous oil and thereafter diluting said concentrate with a very light oil or oily material to form the final grease composition. Many modifications can be made, one of which is to heat-treat the grease concentrate while it is in the gel phase until a recrystallization and consolidation of the soap crystallites into desired fiber form has been attained.

The gel phase may be defined as a two-phase system comprising solid crystalline soap and a liquid phase consisting of either soap-oil jelly or pure oil. This gel phase forms as the grease is cooled from an elevated temperature of above 200° C. at which temperature it is in a jelly phase to a temperature where recrystallization and consolidation of soap crystallites begin to occur, that is, the gel phase. Above 200° C. the soap-oil mixture may be regarded as being in a jelly phase in which soap and oil are in a homogeneous mass.

If the heat-treating step is involved the soap and viscous oil can be admixed and heated to a

temperature of 200–250° C. until a homogeneous mass has been attained. Modifying agents such as antioxidants, corrosion inhibitors, etc. can be added at this point. The homogeneous grease concentrate is then slowly cooled to below its jelly phase but above 100° C. and heat-treated in this temperature range in a quiescent state until the desired soap fiber growth has been attained. This grease concentrate which may contain 10% to 50% soap can be stored, and diluted with a light oil at a convenient time (below about 100° C.) or cooled to room temperature, or preferably to 80° or 90° C. and diluted with a light oil or oily material until the soap content in the final composition varies from 4 to 15% and preferably from 6 to 10%. If desired the finished product can be homogenized prior to being packaged.

By a viscous oil is meant to include an oil having a viscosity of from 500 at 100° F. to 2000 at 100° F. SUS and higher. The oils may be natural or synthetic or mixtures thereof. By light oils is meant to include any suitable liquid having a viscosity below 300 at 100° F. and may include spray oils, gas oil, light petroleum distillates, organic esters, e. g., 2-ethyl hexyl sebacate, butyl acetate, undecyl ricinoleate, butyl oleate, butyl stearate, etc.; low molecular weight alkylene oxide-mono hydroxyl alcohol copolymers, olefin polymers, etc. Generally, oils of 50 to 300 SUS at 100° F. are suitable for use as light oils.

The gelling agents used to form the grease may be soaps of fatty acids and/or their glycerides. The saponifiable material may be fatty acids having from 12 to 32 carbon atoms and they may be saturated, unsaturated or polar-substituted fatty acids, such as capric, lauric, myristic, palmitic, stearic, arachidic, behenic, lignoceric, myristoleic, palmitoleic, oleic, linoleic, cottonseed oil fatty acid, palm oil fatty acids, hydrogenated fish oil fatty acids, ricinoleic, erucic acids and their mixtures and/or their glycerides, such as lard, beef, rapeseed, palm, menhaden, herring oils, etc. Other acids may be included among which are: acids produced by oxidation of petroleum oil and waxes, rosin acids, tall oil acids, abietic acids; naphthenic acids, petroleum sulfonic acids and the like.

A particularly preferred class of saponifiable materials are the hydroxy fatty acids and their glycerides, such as dimethyl hydroxy caprylic acid, dimethyl hydroxy capric, hydroxy phytetoleic acid, ricinoleic acid, ricinelaidic acid, 12-hydroxy stearic acid, 9,10-dihydroxy stearic acid, 4-hydroxy palmitic acid, linusic acid, sativic acid, lanoceric acid, dihydroxy gadoleic, hydroxy behenic acid, quince-oil acid and the like. The preferred hydroxy fatty acids are those in which the hydroxy group is at least 12 carbon atoms removed from the carboxyl group. Also, it is preferable to use hydroxy fatty acids having at least 10 carbon atoms and up to about 32 carbon atoms and preferably those having between 14 and 32 carbon atoms in the molecule. Instead of using the free fatty acids containing a hydroxy radical their glycerides can be used such as castor oil or hydrogenated castor oil or mixtures of free hydroxy fatty acids and their glycerides can be used. Mixtures of hydroxy and non-hydroxy fatty acids can be used to form soaps.

The saponifying agent used to make the soap may be any oxide or hydroxide of one or several of metals selected from Groups I, II, III, IV, VI, VII and VIII. Specifically the cation of the soaps may be represented by Na, K, Li, Cs, Ca,

Sr, Ba, Cd, Zn, Al, Pb and Co. Mixtures of soaps can be used and the soaps can be made in situ or premade soaps can be used to form the grease. Specific examples of preferred soaps and mixtures thereof are: lithium stearate, lithium hydroxystearate, lithium ricinoleate, lithium soap of hydrogenated fish-oil fatty acids, lithium soap of mixed stearic and hydroxystearic acid, sodium stearate, sodium hydroxystearate, sodium oleate, potassium oleate, potassium rosinate, calcium stearate, calcium hydroxystearate, aluminum stearate, aluminum soap of fish-oil fatty acids, barium hydroxystearate, barium stearate, lithium soap of mixed stearic and hydroxystearic acids, lithium soap of mixed oleic and hydroxystearic acid, sodium soap of stearic and hydroxystearic acid; aluminum soap of stearic and oleic acid, aluminum ricinoleate; mixed soaps of lithium stearate and sodium stearate; mixed soaps of lithium hydroxystearate and sodium stearate; mixed soaps of lithium hydroxystearate and calcium stearate, etc. Amine soaps such as triethanolamine oleate can be used in combination with metal soaps or as the gelling agent.

Instead of using only soaps as the gelling agent, mixtures of soaps and other gelling agents, such as organic or inorganic aerogels, e. g. silica aerogels and alumina aerogels can be used.

The soap content of grease compositions of this invention may vary over wide limits and may be as high as 50%. In practice, it is possible by choice of suitable grease-forming lubricant bases to manufacture satisfactory lubricating greases containing only about 10% or less by weight of the soap mixtures. Very satisfactory products are obtained with a total soap content of about 8% by weight on the finished grease.

In order more clearly to set forth the invention, reference is now made to the accompanying drawing, which is a flow diagram illustrating a method of producing grease compositions of this invention. It is understood that modifications as to equipment, its arrangement, type and kind of materials and their proportions can be resorted to without departing from the spirit of the invention as presented in the subjoined claims.

Referring to the flow diagram, the invention can be carried out as follows: Into a slurry tank 1 a pre-made soap or soap mixtures or materials used for making desired soaps and the highly viscous oil are added through lines 2 and 3, respectively. If other additives such as oxidation and corrosion inhibitors, anti-bleeding agent, oiliness agents and the like are added to the grease they can be introduced into the slurry tank 1 through conduit 2 at a convenient time. The oil and soap or soap-forming materials are heated to around 120° C. for a time sufficient to drive out most of the moisture which might be present and is then led through conduit 4, through pump 6 and into heating equipment 9 where the grease is heated to a temperature of from 200° to 250° C. and worked until a homogeneous mixture has been attained. This hot grease concentrate which may contain from 10% to 50% soap may be conducted into a grease storage tank 26 through lines 7 and 28 and at a convenient time conducted through pump 20, and lines 27 and 12 into the final grease-forming kettle 13 where it may be admixed at room temperature or preferably heated to about 90° C. with a light oil (50–300 SUS at 100° F.) which is introduced into a kettle 13 through line 14. The

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amount of light oil used depends upon the concentration of soap desired in the final grease. Generally an amount of light oil is used so that the final soap content should vary from 6% to 15% by weight. If desired the hot grease concentrate may be lead directly into kettle 13 through lines 7, 28, and 15, admixed with light oil from line 14 and worked into the final grease composition.

A preferred embodiment of this invention is to heat-treat the grease concentrate prior to working it into the final grease in kettle or "votator" 13. This is accomplished by regulating the flow of the grease concentrate from heating tank 9 so that by the time it reaches the time or heat-treatment tank 10 the temperature of the grease has dropped below 200° C. but is above 100° C. The grease concentrate is allowed to remain in tank 10 in a quiescent state within the above temperature range for a time sufficient to form the desired fiber length and structure. The heat-treated grease concentrate may then be sent to storage tank 26 or sent to the final grease-forming kettle 13 through pump 11 and line 12 and worked into the desired grease as described above.

The final grease composition may be sent to storage tank 19 through lines 16 and 18 or it may be sent directly to homogenizer 23 at a temperature ranging from room temperature to about 65° C. and then discharged through line 24 into packing container 25. If desired, a portion of the finished grease from kettle 13 can be returned to slurry tank 1 through lines 16 and 29.

To illustrate the process of making greases by this invention, a specific example, namely, the method of making soda base grease will be herein fully described following the flow diagram.

Example 1

Composition of the grease was as follows:

Components	lb./100 lb. Grease
Hydrogenated fish oil acids.....	5.25
Hydrogenated castor oil.....	0.75
Polyethylene glycol having an average molecular weight of 1500.....	0.034
Sodium hydroxide (dissolved in 2 lb. water).....	0.84
Mineral oil (2000 SUS at 100° F.) 19%.....	Balance
Mineral oil (100 SUS at 100° F.) 75%.....	

The acids and glycerides together with the 2000 SUS at 100° F. mineral oil and the caustic solution are introduced into slurry tank 1 and heated at about 80° C. until the fatty materials have melted. The mixture is stirred vigorously to form a slurry and the temperature increased to above 100° C. to drive off the water. During the heating and stirring a modicum of dimethyl silicone can be added if excess foaming occurs and the polyethylene oxide is added through line 2 or 30. The slurry is then lead through conduit 4 and pump 6 into heating tank 9 where the grease concentrate is heated to above 190° C. and preferably at from 204° C. and 225° C. until a homogeneous gel has been attained. The grease concentrate is cooled and introduced into the heat-treatment tank 10 where it is kept at about 160° C. for about 4 hours in a quiescent state. The grease is then conducted through line 21 into storage tank 26 where the grease is allowed to cool to about 90° or 100° C. then it is conducted into grease tank 13 through lines 27 and 12. At a temperature of about 80-100° C. the remaining light mineral oil (100 SUS at 100°

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F. viscosity) is introduced into kettle 13 through line 14 and the entire mass worked until a homogeneous grease has been attained. If desired the grease concentrate can be admixed with the light oil at room temperature. The grease is then lead to homogenizer 23 for working into a homogeneous consistency and texture, after which it is packed in containers 25. Greases compounded by the above method have excellent mechanical stability and are particularly suitable for low-temperature operations.

Example 2

Following the above procedure an excellent grease was prepared in which dioctyl phthalate was used instead of the light mineral oil.

Example 2A

A lithium soap and a mineral oil having a viscosity (Saybolt) of 1000 at 100° F. were introduced into slurry tank 1 and the mixture heated until a homogeneous mass was formed. The slurry was then led through conduit 4 and pump 6 into heating tank 9 where the grease concentrate was heated to around 160-170° C. (1-8 hours) until a homogeneous gel was formed. The grease concentrate was then conducted through lines 7 and 28 into storage tank 20 where the concentrate was cooled to around 100° C. and thereafter conducted into tank 13 through line 27. At a temperature of about 80-100° C., the remaining light mineral oil (100 SUS at 100° F.) was introduced into kettle 13 through line 14 until the soap content varied from 4 to 15% of the total composition and the entire mass was worked until a homogeneous grease had been attained. The grease was then led to homogenizer 23 for working into a homogeneous consistency and texture, and then packed in containers 25.

Example 3

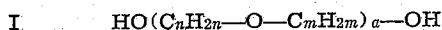
A soda base grease composed of the ingredients of Example 1 was prepared by a different method from that described in Example 1 and the result was a much poorer grease. This grease was prepared by admixing the soap ingredients, stabilizer and all of the mineral oil (which had a blended viscosity of 150 SUS at 100° F.) in slurring tank 1. The slurry was then lead to heating tank 9, heat-treatment tank 10 and then into homogenizer 23 for working and then packaged.

The three greases were subjected to the Shell roll stability test in order to determine their mechanical stabilities. The roll stability tester is a simulated roller bearing and consists of a 90 mm. I. D. x 180 mm. long cylinder inside of which is a 5 kg. weighted roller, 60 mm. diam. x 176 mm. long. The grease to be tested is placed in the free space and the outer cylinder is rotated on its axis at 160 R. P. M. The grease is worked by rolling action of the inner cylinder. The stability of a grease is expressed as hours of rolling before its breakdown, and the samples prepared in the three examples behaved as follows:

Composition	Roll Test (hr.)
Example 1.....	400
Example 2.....	400
Example 3.....	6

To improve grease compositions made by the present method minor amounts of stabilizing agents can be added to the grease.

Particularly desirable stabilizing agents which can be used with grease compositions of this invention are the alkylene glycol and/or alkylene thioglycol polymers as well as their mono-esters and other polymeric derivatives. The alkylene glycol polymeric materials can be represented by the following general structural formula:



wherein m and n and a are integers. Preferably the polymeric alkylene glycols as represented by the above general formula should be such that the factor a times the number of carbon atoms within the brackets should be at least 6 and more.

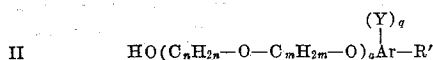
The polymeric alkylene glycols can be made by polymerizing in the presence of a catalyst such as iodine, hydriodic acid and the like, ethylene, propylene, isobutylene, n -butylene oxides and/or their mixtures. Such polymers can also be produced by reacting a monohydric alcohol with an alkylene oxide. Thus a suitable product can be made by reacting n -butanol with propylene oxide at between about 212° to 230° F. under pressure and in the presence of an alkali catalyst.

The polymerized higher polyalkylene glycols having between 2 and 6 carbon atoms in the alkylene group are most effective as additives of this invention and those containing the ethylene and propylene groups are preferred. The average molecular weight of the polyalkylene glycols may be from about 200 to about 7000 and the preferred molecular weight being from about 1000 to 6000.

It is desired to point out that the higher polyalkylene glycols are composed of mixtures of several polymers, for example, a polyethylene glycol having an average molecular weight of 400 consists of various glycols varying from a minor amount of monethylene glycol and increasing up to the pentadecaethylene glycol. Therefore, it is the average molecular weight which is specified and wherein the present specification, polyalkylene glycols or polyethylene glycols are referred to, they define the higher glycols having an average molecular weight in excess of 200 and preferably in excess of 400, those with an average molecular weight of between about 1000 to 1500 being very effective in carrying out the present invention.

In lieu of the polyalkylene glycols, the ester and other derivatives can be used. The esters can be made from a variety of acids having between 1 to about 22 carbon atoms and preferably between about 10 to 18 carbon atoms. Acids which may be used are the aliphatic, aromatic, cyclic, sulfonic acids and the like. Fatty acids and especially the higher fatty acids are preferred and include such acids as lauric, myristic, palmitic, stearic, arachidic, behenic, oleic, ricinoleic, hydroxystearic, phenylacetic, phenyl stearic acids and the like. However, such acids as naphthenic acid, oil-soluble petroleum sulfonic acids, tall oil fatty acids, aromatic acids, e. g. salicylic and phthalic acids and the like may be used to form the esters. Specific examples of esters of this type are the polyethylene glycol monostearate, polyethylene glycol monooleate and the like.

Ether derivatives of polyalkylene glycols may be made by any conventional method and the aromatic ethers of polyalkylene glycols having the general formula



wherein Ar is an aromatic radical having attached thereto at least one alkyl radical denoted by R' having from 1 to about 8 carbon atoms and wherein Y is a fatty acid derivative, n , m and a are integers as in I and q is a number selected from the group consisting of zero or 1.

Minor amounts of oxidation inhibitors can be added to grease compositions of this invention such as N -butyl paraphenylene diamine. Also effective as oxidation inhibitors are alpha or beta naphthylamine, phenyl-alpha or -beta naphthylamine, alpha-alpha, beta-beta, or alpha-beta dinaphthylamine, diphenylamine, tetramethyl diamino diphenylmethane, petroleum alkyl phenols, and 2,4-ditertiary butyl 6-methyl phenol.

Corrosion inhibitors which are particularly applicable with compositions of this invention are N -primary amines containing at least 6 and more than 18 carbon atoms in the molecule such as hexylamine, octylamine, decylamine, dodecylamine, octadecylamine, heterocyclic nitrogen containing organic compounds such as alkyl substituted oxazolines and oxazoline salts of fatty acids and nitrophenol.

Extreme pressure agents can be added to such grease and the preferred agents comprise esters of phosphorus acids such as triaryl, alkylhydroxy, aryl, or aralkyl phosphates, thiophosphates or phosphites, etc., neutral aromatic sulfur compounds such as diaryl sulfides and polysulfides, e. g. diphenyl sulfide, dicresol sulfide, dibenzyl sulfide, methyl butyl diphenol sulfide, etc., diphenyl selenide and diselenide, dicresol selenide and polyselenide, etc., sulfurized fatty oils or esters of fatty acids and monohydric alcohols, e. g., sperm oil, jojoba oil, etc., in which the sulfur is tightly bound; sulfurized long-chain olefins obtained by dehydrogenation or cracking of wax; sulfurized phosphorized fatty oils, acids, esters and ketones, phosphorus acid esters having sulfurized organic radicals, such as esters of phosphoric or phosphorus acids with hydroxy fatty acids, chlorinated hydrocarbons such as chlorinated paraffins, aromatic hydrocarbons, terpenes, mineral lubricating oils, etc.; or chlorinated esters of fatty acids containing the chlorine in positions other than the alpha position.

Additional ingredients which can be added are anti-wear agents such as oil-soluble urea or thiourea derivatives, e. g. urethanes, allophanates, carbazides, carbazones, etc.; or rubber, polyisobutylene, polyvinyl esters, etc.; VI improvers such as polyisobutylene having a molecular weight above about 800, voltolized paraffin wax, unsaturated polymerized esters of fatty acids and monohydric alcohols, etc.; oiliness agents such as stearic and oleic acids and pour point depressors such as chlorinated naphthalene to further lower the pour point of the lubricant.

The amount of the above additives can be added to grease compositions of this invention in around about 0.01% to less than 10% by weight and preferably 0.1% to 5.0% by weight.

Greases of this invention are applicable for general automotive uses, and are excellent aircraft greases, industrial greases and the like.

We claim as our invention:

1. The method of making a wide temperature grease composition comprising admixing an oil having a viscosity above 500 at 100° F. SUS with a grease-forming gelling agent in an amount so that the gelling agent comprises from 10% to about 50% of the mixture, heating until a homogeneous non-crystalline mass has formed, cooling said grease concentrate to a temperature

below 100° C., and admixing into said grease concentrate at this temperature a light oil having a viscosity below 300 at 100° F. SUS until the content of the gelling agent based on the total composition ranges from about 5% to about 15% by weight.

2. The method of making a wide temperature grease composition comprising admixing an oil having a viscosity above 500 at 100° F. SUS with a grease-forming gelling agent in an amount so that the gelling agent comprises from 10% to about 50% of the mixture, heating said mixture to a temperature above 200° C. until a homogeneous non-crystalline mass has formed, cooling said grease concentrate to a temperature below 100° C. and admixing into said grease concentrate at this temperature a light oil having a viscosity below 300 at 100° F. SUS until the content of the gelling agent based on the total composition ranges from about 5% to about 15% by weight.

3. The method of making a wide temperature grease composition comprising admixing an oil having a viscosity above 500 at 100° F. SUS with a grease-forming soap in an amount so that the soap comprises from 10% to about 50% of the mixture, heating said mixture to a temperature above 200° C. until a homogeneous non-crystalline mass has formed, cooling said grease concentrate to a temperature below 100° C. and admixing into said grease concentrate at this temperature a light oil having a viscosity below 300 at 100° F. SUS until the content of the soap based on the total composition ranges from about 5% to about 15% by weight.

4. The method as defined by claim 2 in which a small amount of an oxidation inhibitor is incorporated into the grease concentrate.

5. The method as defined by claim 2 in which the grease concentrate is heat-treated in a quiescent state prior to being cooled to below 100° F. for dilution with a light viscosity oil.

6. The method of making a wide temperature grease composition comprising admixing an oil having a viscosity above 500 at 100° F. SUS with a grease-forming gelling agent in an amount so that the gelling agent comprises from 10% to about 50% of the mixture, heating said mixture until a homogeneous non-crystalline mass has formed, reducing the temperature to a point where recrystallization occurs but not below 110° C. and maintaining the grease concentrate in this temperature range for from about 1 to 8 hours, cooling thereafter said grease concentrate to below 100° C. and diluting said grease with a light oil having a viscosity at 100° F. of from 75 to 300 SUS at 100° F. until the soap content of the grease varies from 4 to about 15% by weight.

7. The method of making a soda soap grease which comprises forming a grease concentrate containing 10% to 50% of soda soap in a highly viscous oil having a viscosity at 100° F. of 500 to 1000 SUS, maintaining said grease concentrate at a temperature of from about 160 to about 170° C. for from about 1 to 8 hours, cooling thereafter said grease concentrate to below 100° C. and diluting said grease with a light oil having a viscosity at 100° F. of from 75 to 300 SUS until the soap content of the grease varies from about 4% to about 15% by weight.

8. The method of making a wide temperature grease composition comprising admixing an oil having a viscosity above 500 at 100° F. SUS with 10% to 50% soda soap and from 0.01% to about 1% polyalkylene glycol and heating said mixture

to a temperature above 200° C. until a homogeneous mass has formed, reducing the temperature to about 160° and 170° C. and maintaining said grease in this temperature range from about 1 to 8 hours, cooling thereafter said grease concentrate to below 100° C. and diluting said grease with a light oil having a viscosity at 100° F. of from 75 to 300 SUS at 100° F. until the soap content of the grease varies from 4% to about 15% by weight.

9. The method of making a lithium soap grease which comprises forming a grease concentrate containing 10% to 50% of lithium soap in a highly viscous oil having a viscosity at 100° F. of 500 to 1000 SUS, maintaining said grease concentrate at a temperature from 160 to about 170° C. for from 1 to 8 hours, cooling thereafter said grease concentrate to below 100° C. and diluting said grease with a light oil having a viscosity at 100° F. of from 75 to 300 SUS until the soap content of the grease varies from about 4% to about 15% by weight.

10. The method of making a soda soap grease which comprises forming a grease concentrate containing 10% to 50% of soda soap in a highly viscous oil having a viscosity at 100° F. of 500 to 1000 SUS, maintaining said grease concentrate at a temperature of from 160 to about 170° C. for from 1 to 8 hours, cooling thereafter said grease concentrate to below 100° C. and diluting said grease with dioctyl phthalate until the soap content of the grease varies from about 4% to about 15% by weight.

11. The method of making a lithium soap grease which comprises forming a grease concentrate containing 10% to 50% of lithium soap in a highly viscous oil having a viscosity at 100° F. of 500 to 1000 SUS, maintaining said grease concentrate at a temperature of from about 110 to 160° C. for from 1 to 8 hours, cooling thereafter said grease concentrate to below 100° C. and diluting said grease with dioctyl phthalate until the soap content of the grease varies from about 4% to about 15% by weight.

12. The method of making a soda soap grease which comprises forming a grease concentrate containing 10% to 50% of soda soap and from 0.01% to about 1% polyalkylene glycol in a highly viscous oil having a viscosity at 100° F. of 500 to 1000 SUS, maintaining said grease concentrate at a temperature of from 160 to about 170° C. for from 1 to 8 hours, cooling thereafter said grease concentrate to below 100° C. and diluting said grease with a light oil having a viscosity at 100° F. of from 75 to 300 SUS until the soap content of the grease varies from about 4% to about 15% by weight.

13. The method of making a lithium soap grease which comprises forming a grease concentrate containing 10% to 50% of lithium soap and from 0.01% to 1% polyalkylene glycol in a highly viscous oil having a viscosity at 100° F. of 500 to 1000 SUS, maintaining said grease concentrate at a temperature of from about 160 to 170° C. for from 1 to 8 hours, cooling thereafter said grease concentrate to below 100° C. and diluting said grease with a light oil having a viscosity at 100° F. of from 75 to 300 SUS until the soap content of the grease varies from about 4% to about 15% by weight.

14. The method of making a soda soap grease of hydroxy fatty acids of high molecular weight which comprises forming a grease concentrate containing around 50% soap and from 0.01 to about 1% polyalkylene glycol in a highly viscous

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mineral oil having a viscosity at 100° F. of around 2000 SUS, maintaining said grease concentrate at a temperature of from 160° to about 170° C. for around 8 hours, thereafter cooling said grease concentrate to below 100° C. and diluting said grease with a light mineral oil having a viscosity at 100° F. of around 100 SUS until the soap content of the grease is around 8% by weight.

15. The method of making a soda soap grease which comprises forming a grease concentrate containing 10% to 50% of soda soap in a highly viscous oil having a viscosity at 100° F. of 500 to 2000 SUS, maintaining said grease concentrate at a temperature of from about 160° to about 170° C. for from about 1 to 8 hours, thereafter cooling said grease concentrate to below 100° C. and diluting said grease with a light oil having a viscosity at 100° F. of from 75 to 300 SUS until the soap content of the grease is from about 4% to about 15% by weight.

16. The method of making an alkali metal soap grease which comprises forming a grease concentrate containing 10% to 50% of alkali metal soap in a highly viscous oil having a viscosity at 100° F. of 500 to 2000 SUS, maintaining said grease concentrate at a temperature of from about 160° to about 170° C. for from about 1 to 8 hours, thereafter cooling said grease concen-

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trate to below 100° C. and diluting said grease with a light oil having a viscosity at 100° F. of from 75 to 300 SUS until the soap content of the grease is from about 4% to about 15% by weight.

17. The method of making soda soap grease by method of claim 14 in which the soda soap is made from a mixture of hydrogenated fish oil acids and hydrogenated castor oil, said hydrogenated fish oil acids being present in a predominant amount.

18. The method of making soda soap grease by the method of claim 15 in which the soda soap is made from a mixture of hydrogenated fish oil acids and hydrogenated castor oil, said hydrogenated fish oil acids being present in a predominant amount and in which the light oil having a viscosity at 100° F. of from 75 to 300 SUS is dioctyl phthalate.

ROBERT J. MOORE.

JOHN NORTON WILSON.

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