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2,394,567

LUBRICATING GREASE COMPOSITION

Filed Dec. 17, 1941

2 Sheets-Sheet 1

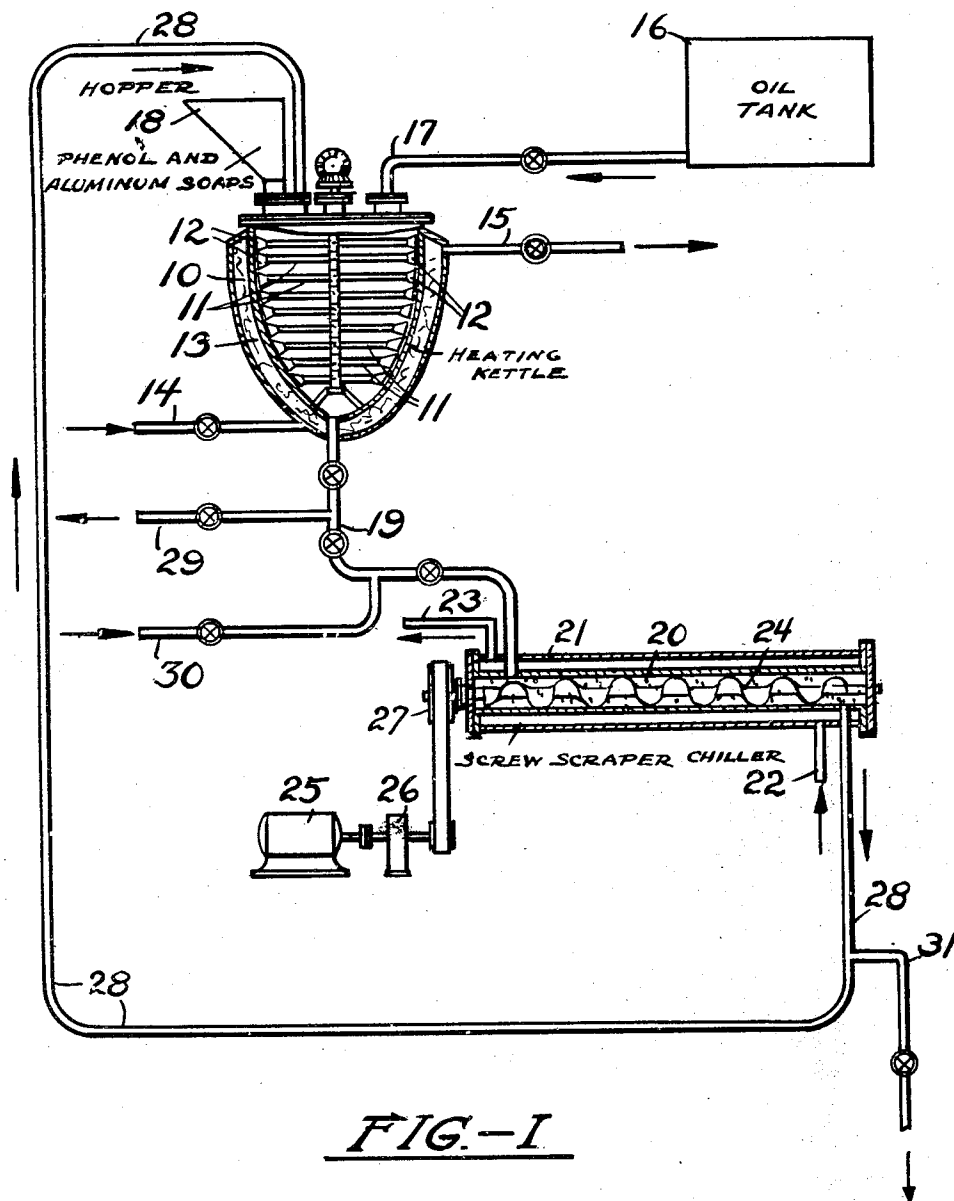


FIG.-I

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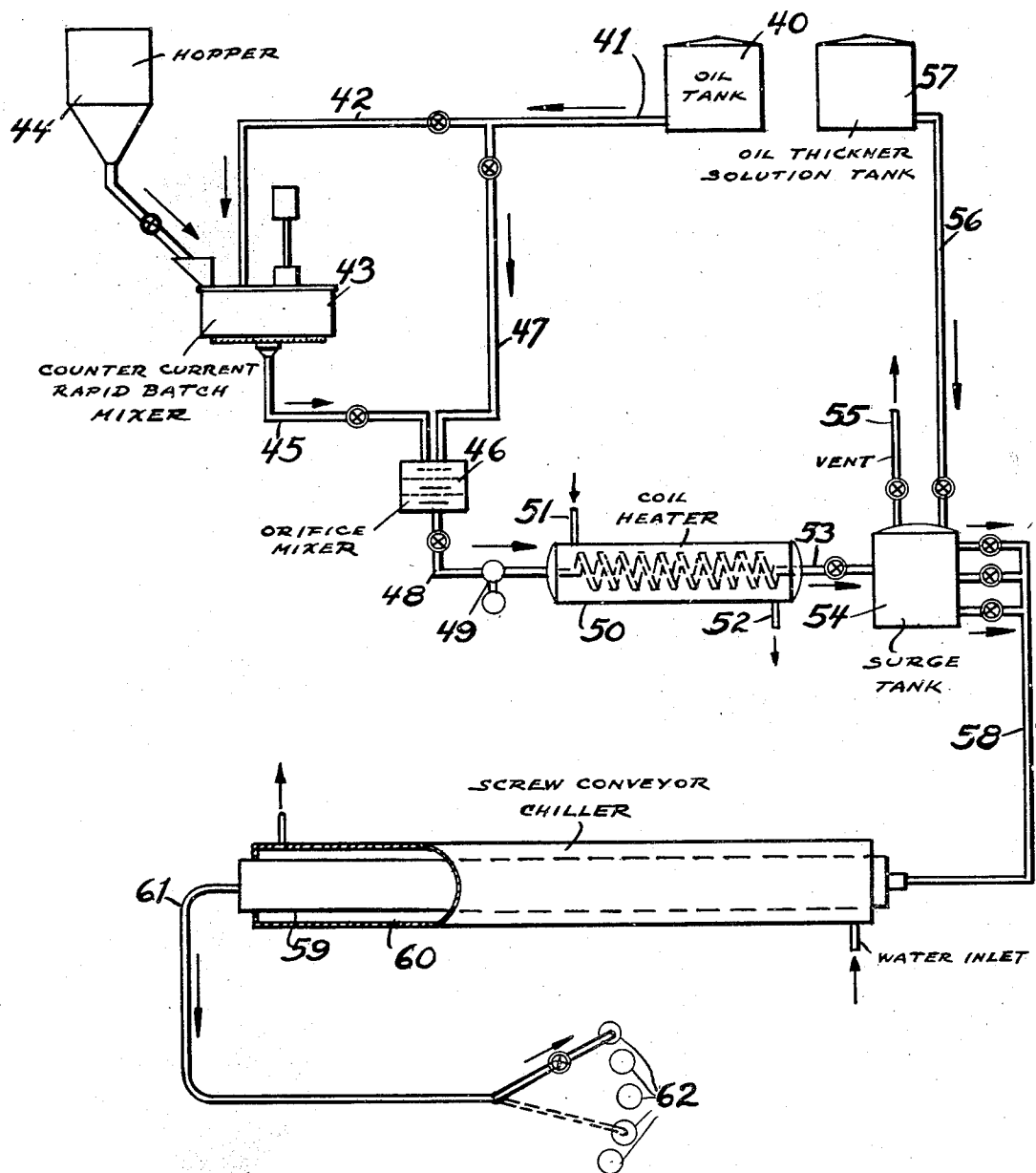


FIG. -II

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UNITED STATES PATENT OFFICE

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LUBRICATING GREASE COMPOSITION

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Standard Oil Development Company, a corporation of Delaware

Application December 17, 1941, Serial No. 423,276

14 Claims. (Cl. 252—37)

This invention relates to aluminum soap greases highly advantageous in the lubrication of machinery operating under conditions of local high temperature increases, of irregular and shock load effects, and of frictional forces beyond the film strength of any lubricating oil base material. The invention relates more particularly to aluminum soap greases adapted for the lubrication of industrial machinery and the chassis parts of automobiles and crawler type tractors and to an improved process for the preparation of such greases.

Aluminum soap greases have proven to be outstanding lubricants for many types of industrial machinery and especially for the lubrication of automotive and tractor chassis parts. Difficulties, however, in the manufacture of aluminum soap greases have greatly hampered their production and utilization. It is well known for example that when aluminum stearate is dispersed in a mineral oil at an elevated temperature and the product cooled, compositions unsuitable as lubricants are obtained. The compositions, for example, vary in texture from being stringy, viscous liquids to solids having a hard and grainy structure. However, when the soap dispersions are carefully cooled in small quantities, a change in structure usually occurs and there is obtained a firm, transparent gel suitable as a lubricant. On the manufacturing scale attempts therefore to cool quickly bulk quantities of aluminum soap-oil blends have resulted in the formation of physically heterogeneous mixtures unsuitable for lubrication purposes. Effecting the rapid cooling in thin layers is unsatisfactory because often times there are formed soft granular masses unsuitable as grease type lubricants. Thus, on the commercial scale it has been found necessary to undertake the costly and laborious process of running the hot grease composites after blending into shallow pans then allowing the mass to cool slowly, then passing the mixture to suitable equipment for reworking and then filtering to effect the removal of any granular surface films and to obtain a relatively homogeneous composition.

Attempts to cool grease composites in large quantities after blending have shown that the mixtures can be cooled satisfactorily from compounding temperatures of about 300° F. to between about 140° F. and 160° F. only. After cooling to a temperature within this range the change

in structure may occur from being a viscous, gummy liquid to that of being a suitable gel. This change, however, occurs only when the cooling period is between eight and sixteen hours and when the mass is cooled in small quantities without agitation. However, in the process of cooling large quantities of grease a portion at the outer surface rapidly changes to a gel, while the rest of the mass has not attained the necessary temperature for the transition to suitable gel formation. Under these processing conditions the outer layers readily break down due to excessive growth of soap particles, and thereafter form thin, liquid masses unsuitable as lubricants.

It has now been found that when small quantities of phenolic compounds, such as the simple phenols, alkylated phenols, naphthols and the like are incorporated in blended mixtures of aluminum stearate and mineral oil, composites in bulk can be cooled rapidly with agitation to temperatures as low as 85° F. and in layers of about 1/4" thick to temperatures as low as 50° F. without granulation or decomposition. The use in this manner of small quantities of phenolic compounds thus greatly expedites the manufacture of aluminum soap greases. Furthermore, this use of phenolic compounds permits the preparation of aluminum soap greases on a large scale, continuous basis, and facilitates the packaging of the grease directly from the cooling devices into large shipping containers, without reworking or filtering. These advantageous effects of phenolic compounds have been found to occur without the development of any disadvantageous effects upon any of the desirable lubricating properties of aluminum soap greases. Moreover, these advantageous effects of phenolic compounds in aluminum soap greases are not disadvantageously affected by the presence of accessory additive materials such as oil thickening agents, anti-corrosive and anti-oxidation agents.

Thus the present invention relates to the preparation of highly stabilized aluminum soap grease compositions containing a phenolic compound as the special additive material for a grease mixture of suitable mineral oil base and an aluminum soap of a saturated fatty acid containing between 10 and 20 carbon atoms in the molecule, such as aluminum stearate. The amount in which the phenolic compound is present in the compositions varies usually between 5% and 30% by weight of the amount of the aluminum soap. The alu-

minum soap is blended with the mineral oil to form a grease of suitable composition in amounts between 3% and 20% but usually between about 3% and 10% by weight of the oil. Accessory constituents to impart to the composites other desirable characteristics include free, unsaturated fatty acids containing 10 and 20 carbon atoms in the molecule and their aluminum soaps, naphthenic acids and their aluminum soaps; and oil thickening agents, such as latex or polybutene.

Phenolic compounds in general have been found to be effective as additives to grease compositions according to the present invention. In the choice of a phenolic compound as a suitable blending agent for a particular grease composition, selection is made largely on the basis of its vapor pressure and its relative effects upon oil solubility and miscibility with other constituents as regards effecting the lowering of the transition temperature. It is also important to select the phenolic compound on the basis of its effect in physically stabilizing the finished grease. Thus it has been found that the mono nuclear phenols are in general slightly more effective than the poly nuclear phenolic compounds, and that for vapor pressure and solubility characteristics it is preferable to employ the alkylated phenolic compounds. In regard to the physical stabilization of the grease products certain of the substituted phenols such as the amino phenols are preferable. However, substituted phenols such as the halogenated and sulfur-bearing derivative are effective. Particularly effective phenols are the alkylated compounds having one or more alkyl side chains. Of the various phenolic compounds effective for purposes of this invention particular mention may be made of naturally occurring phenols, derived especially from petroleum and coal tar, the cresols, the amyl phenols, dihydric phenols, α -naphthol and β -naphthol.

The effect of the phenolic compounds appears to be connected pre-eminently with the compounding and cooling steps in the preparation of the greases. It appears that phenolic compounds function in the aluminum soap-mineral oil blends to permit rapid cooling without crystallization of the aluminum soaps. Thus, the phenolic compounds permit the mixture after compounding to be agitated during cooling and the transition in structure to occur at the lower temperatures than hitherto in vessels of even 400 lbs. capacity.

In preparing the grease compositions of this invention, the oil employed is preferably derived from a naphthenic base crude, as for example oils of the Coastal type. The viscosity of the oil is usually above about 85 seconds Saybolt at 210° F., and preferably from 50 to 200 seconds Saybolt at 210° F. The aluminum soap and the phenolic compounds are added to the oil at a temperature between about 130° F. and 150° F. The heating may be carried out in a coil type heater or in the usual type kettle. A suitable heating kettle is furnished with mechanical means of agitation, usually paddles having at the outer sweeps close fitting scraper adjustments to pass over its inner surface in order to insure good heat transfer and to obviate the development of surface films. Usually the aluminum soap and the phenolic compounds are blended with about 10% to 20% of the total quantity of the oil to be used and the mixture worked into a thick non-lumpy paste. This paste is then stirred into the balance of the oil, and heated to a temperature of between 280° F. and 300° F. and held at that temperature

until all of the soap and phenolic compounds are thoroughly incorporated to form a smooth homogeneous mass. The mass after heating is then rapidly cooled by passing through cooling equipment to a temperature of about 90° F. The cooling may be effected by passing the material into shallow pans and then returning the cool grease to mixing equipment wherein the mixture is allowed to settle to about the transition temperature. On the other hand, the material may be rapidly cooled by passing through any type of jacketed equipment and preferably through a helical screw conveyor type cooler.

Another means of preparing the grease is to first form a slurry with a small quantity of the oil base material and the aluminum soap and phenolic compounds, and then to pass the slurry through an orifice mixer to which is also supplied the further quantities of oil as are required to prepare the finished grease. The mixture is then passed through a coil heater at a temperature of about 300° F. and then allowed to settle to permit the separation of any occluded gases before passing through a helical screw type cooler. By this means a grease can be satisfactorily prepared in large quantities on a continuous basis, and the product directly passed to the shipping containers from the cooling equipment.

An illustration of the method of preparing the grease involving the use of the usual type kettle for heating and blending the constituents is afforded by the following description which should be read in conjunction with the drawings presented as Figure 1.

Figure 1 shows a jacketed kettle 10 fitted internally with paddles 11 having scraper terminals 12 which can be finely adjusted to work upon the entire surface of the kettle. The motion of the paddles in the kettle insures very complete and thorough agitation within the kettle. The jacket portion 13 of the kettle 10 is adapted for the passing in through line 14 and the passing out through line 15 of steam either under normal or superatmospheric pressure as temperatures within the kettle 10 require. Under the conditions of such means of agitation and of a temperature between about 280° F. and about 300° F., sufficient oil of suitable character as a mineral oil base for the preparation of the grease composition is supplied from storage tank 16 to the kettle 10 through line 17 and admixed with the aluminum soap and phenolic compounds supplied through opening 19 to make a smooth thick paste. Further quantities of oil to make the grease of desired consistency are then supplied through line 17 and the mass agitated until a smooth homogeneous composition is obtained.

When the mass in the kettle has become smooth and homogeneous, the mixture is passed from the bottom of the kettle through line 19 to a screw scraper chiller 20. The chiller 20 has an outer jacket 21 through which a cooling medium, usually water, passes in through line 22 and out through line 23. The screw 24 within the chiller is usually operated countercurrently to the direction of the flow of the grease from line 19 and countercurrently to the direction of the cooling fluid in the jacket 21. The scraper screw is operated by the motor 25 through gears 26 and transmission 27. The cooled grease passes from the chiller through line 28.

In batch operation the cooled grease is recycled through line 28 to the kettle 10. By such recycling the entire quantity grease composition is brought to about the transition temperature at

about the same time. When this temperature within the mass is reached, agitation within the kettle is discontinued and the mass is allowed to settle and to cool further until the transition in structure has been satisfactorily effected. The gel composition is then removed from the kettle through a T 29 on line 19 and passed to shipping containers.

In continuous operation it is usual to employ a series of kettles similarly equipped to that designated by the numeral 10 and to pass the hot mixture to the chiller in a relatively continuous stream from the various kettles through the T 30 on line 19. In this manner of operation the cooling effected in the chiller 20 is more complete than that in batch operation, namely the cooling in the chiller is effected to about the transition temperature. The cooled grease in such operation is passed directly through a T 31 on line 28 to the shipping containers wherein settling and further cooling to atmospheric temperature are effected.

Figure 2 indicates a second processing method by which the grease can be prepared. According to this process the oil is passed from a supply tank 40 through lines 41 and 42 into a counter-current rapid batch mixer 43. To the mixer 43 is also added through the hopper 44 the required amounts of aluminum soap and phenolic compounds. The oil, the aluminum soaps and the phenol are added in quantities so as to form a free flowing slurry composition. The slurry is then passed through line 45 to an orifice mixer 46, to which is also supplied through line 47 the further quantities of oil from the supply 40, to form the grease of the desired composition. The mixture is then passed through line 48 and pump 49 to the coil heater 50 wherein it is heated to the necessary compounding temperature which is usually about 300° F. by means of steam passed through lines 51 and 52. The hot mixture then passes through line 53 to a surge tank 54 wherein any occluded air or gas formed during the heating process is permitted to escape through the vent pipe 55. To the surge tank may also be supplied through line 56 an oil thickening agent, such as polybutene or latex, from the tank 57. The grease mixture is removed from the surge tank 54 through the line 58 and passed to the screw conveyor type chiller 59, which is cooled by the water jacket 60. The grease is cooled usually therein to a temperature of about 90-100° F. and then passed directly through pipe 61 to the shipping containers 62.

The following example is presented to illustrate the preparation and various characteristics of suitable greases prepared according to this invention:

A grease of the following composition was prepared:

	Per cent
Aluminum stearate	6.50
Tert.-octyl phenol	0.75
Lubricating oil of 200 S. U. vis./210° F.	14.00
Polybutene	0.25
Lubricating oil of 70 S. U. vis./210° F.	78.50

The lubricating oil of 200 S. U. vis./210° F. and the aluminum stearate were charged to a heating kettle as illustrated in Figure 1, and the mixture agitated by means of the paddles for about 15 minutes. Then the lubricating oil of 70 S. U. vis./210° F. preheated to about 130° F. was added and agitation continued. The tert.-octyl phenol and polybutene were then added and the temperature of the mixture raised to about 280° F.

At this point the heating was discontinued and the mixture cooled by passing cold water through the kettle jacket. The grease was thus cooled with agitation to about 95° F., then agitation discontinued and the mass allowed to stand for 2½ hours. The grease after this period of standing was found to be of a smooth plastic gel nature. The grease so prepared had an unworked penetration at 90° F. of 255, and a worked penetration at 90° F. of 338.

This grease was then heated to 310° F. and held at that temperature for 30 minutes. The grease was cooled to 305° F. by circulating cold water through the kettle jacket and then pumped into the cooler supplied with water at 72° F. The grease after one pass through the cooler (temperature of exit water 88° F.) was passed into various containers at different temperatures and allowed to cool therein to atmospheric temperature. The following table presents the results:

Amount and capacity of container	Temperature, °F.	Worked penetrations 24 hours later	
		Outer layers	Core
100 lb. keg	160-166	Liquid	Liquid.
25 lb. pail	142	342	Semi-liquid.
Do.	120	342	380.
Do.	110	353/355 1	346/350 1
Do.	106	338/344 1	342/349 1
5 lb. can	106	349	336/331 1
100 lb. keg	106	349	345.
400 lb. drum	95	350	353.

1 The second value was obtained twelve days later than the first value.

Comparison of these data demonstrates that cooling to relatively low temperatures, then allowing the masses to stand, even with amounts of 100 lbs. and 400 lbs. can be employed in the preparation of aluminum stearate greases containing tert.-octyl phenol.

In a second testing of the grease composition a sample was heated to 300° F. over a period of about 80 minutes and heating immediately discontinued. The grease at 300° F. was circulated over a prolonged period through the cooler into kettle with the following results:

Time	Temperature, °F. at end of cooler	Temperature in kettle, °F.
1 minute	159	300
1 hour 45 mins	121	148
2 hours	119	144
2 hours 20 mins	115	135
2 hours 30 mins	114	131
2 hours 35 mins	113	130

The grease cooled to 113° F. was then passed at the rate of 16 lbs./minute to fill a 400 lb. barrel. After standing for 20 hours at atmospheric conditions the core was found to have a temperature of 110° F. and the following penetration data were obtained.

	Outer layers	Core	Representative sample of same material cooled in 1 pound can
Unworked penetration	261	310	235
Worked penetration	356	400+	335

Comparison of these data demonstrates the effect of the rate of cooling in the presence of tert.-octyl phenol of aluminum stearate greases

in two widely different size containers, and that the penetration values of the outer layers of cooled bulk masses approximate that of the product cooled in smaller size vessels. When a sample of the grease composition of Example I was passed directly from the kettle at 300° F. to fill a 400 lb. drum and the mass allowed to stand for 10 days, the product was a semi-fluid mass wholly unsuitable as a grease-type lubricant. On the other hand when a similar batch of grease was cooled in the kettle to 95° F. and then passed into a 400 lb. drum, the product was a satisfactory grease product, having no grain formation, and the following penetration values:

	Rim	Center
Unworked.....	261	269
Worked.....	353	350

Thus, it has been shown that satisfactory aluminum soap greases can be prepared in the presence of phenolic compounds from blends of the soaps of the saturated fatty acids containing between 10 and 20 carbon atoms in the molecule, such as aluminum stearate, in viscous oils by rapidly cooling from the compounding temperatures to almost normal temperatures. This rapid cooling to atmospheric temperatures permits the grease products to be packaged directly into bulk shipping containers. The discovery of this effect of phenolic compounds obviates the time- and labor-consuming and the costly prior art process of preparing aluminum soap greases by pan-cooling and subsequent reworking, and also avoids the consistency loss based upon the quantity of aluminum soap employed. It would seem that the action of the phenolic compounds has basis in preventing the recrystallization of the aluminum soaps at temperatures below about 140° F., that is, an effect in grease compositions similar to that of pour depressants in waxy mineral oils inhibiting the crystallization of the wax content. Thus, the use of phenolic compounds permits departure from the prior art procedure of being able to cool the grease to minimum temperatures of about 140° F. and then having to continue the preparation of the grease by pan-cooling, by allowing the hot grease mixture to be cooled with agitation to temperatures well below 140° F. In this manner greases having a worked penetration at 77° F. of between about 50 and about 360 or higher may be easily and readily prepared.

The advantages of the use of phenolic compounds in the preparation of aluminum soap greases are attained without loss of any of the desirable properties of aluminum soap-mineral oil grease composites. It would seem that in the grease products containing the phenolic compounds, the advantageous relationship between the various constituents insuring good lubricating quality is attained and that in addition there is exerted an added stabilizing effect. In other words, it appears that a minor portion of the composition exists as a colloidal dispersion of the soap compounds in a relatively stable emulsion of a major portion of a colloidal dispersion of other soap compounds in the mineral oil of suitable consistency. This relationship, as indicated by tests, such as penetration, adhesiveness to metal surfaces, internal cohesion, melting point, etc., is considered important in order that there be formed on the bearing surfaces a satisfactory adhesive lubricating film while the colloidal phase functions to offer substantial resist-

ance toward heat conduction from the surface film into the mass thereby minimizing the effect of friction, speed and load during service upon the bulk of the grease composition.

The present invention is not to be limited by any theory of manufacture or to any particular type of aluminum soaps, but only to the following claims or their equivalent.

What is claimed is:

1. Process for preparing aluminum soap greases which comprises thoroughly mixing at a temperature from about 280° F. up to about 300° F. a viscous mineral lubricating oil, about 3 to 10% of aluminum stearate and from about 0.25% to 1.5% of a phenolic compound, rapidly cooling the heated mixture to a temperature below 140° F. and allowing the mass to stand.
2. Process according to claim 1 in which the phenolic compound is a mono-nuclear phenol.
3. Process according to claim 1 in which the phenolic compound is an alkyl phenol having between 3 and 8 carbon atoms in the alkyl grouping.
4. Process according to claim 1 in which the phenolic compound is tert.-octyl phenol.
5. Process according to claim 1 in which the thorough mixing at an elevated temperature is effected by passing the ingredients first through an orifice mixer and then through a coil heater.
6. Process for preparing aluminum soap greases which comprises thoroughly mixing at a temperature from about 280° F. up to about 300° F. a viscous mineral lubricating oil, about 3 to 10% of aluminum stearate and from 0.25% to 1.5% of a mono-nuclear phenolic compound, rapidly cooling the heated mixture to a temperature below about 140° F., in a flowing stream and allowing the cooled mass to stand.
7. Process for preparing aluminum soap grease according to claim 6 in which the rapid cooling is effected by passing through a helical screw scraper type cooling equipment in which the flow of the heated mixture is in the opposite direction to that of the motion of the screw and countercurrent to the flow of the fluid in the cooling jacket.
8. Process according to claim 6 in which the mono-nuclear phenolic compound is an alkylated mono-nuclear phenol.
9. Process for preparing semi-fluid aluminum soap greases for the lubrication of chassis parts of crawler-type tractors which comprises thoroughly mixing at a temperature from about 280° F. up to about 300° F. a viscous mineral lubricating oil of between 150-200 seconds Saybolt at 210° F., about 1 to 10% of aluminum stearate and from 0.25% to 1.5% of an alkylated mono-nuclear phenolic compound, rapidly cooling the heated mixture in a flowing stream to a temperature below about 140° F., and then allowing the mixture to stand.
10. A lubricating grease for the chassis parts of crawler-type tractors comprising a mineral lubricating oil having a viscosity of from about 150 to 200 seconds Saybolt at 210° F., about 5% aluminum stearate, 0.75% of an alkylated mono-nuclear phenol, about 0.1% polybutene, the whole being worked into a homogeneous mass having a worked penetration at 77° F. of about 325.
11. A lubricating grease according to claim 10 in which the alkylated mono-nuclear phenol is tert.-octyl phenol.
12. Process for preparing an aluminum soap grease which comprises thoroughly mixing at an

elevated temperature sufficient to effect substantially homogeneous blending a viscous mineral lubricating oil with at least 3% of an aluminum soap of a saturated fatty acid containing at least 10 carbon atoms per molecule and from about 0.25 to 1.5% of phenolic compound, and rapidly cooling the entire mass in bulk without agitation to a temperature below 140° F.

13. Process according to claim 12 in which the phenolic compound is an alkyl phenol having from 3 to 8 carbon atoms in the alkyl grouping.

14. Process according to claim 12 in which the phenolic compound is tert.-octyl phenol.

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