Sept. 21, 1948. A. BEERBOWER ET AL 2,449,580

LUBRICATING GREASE COMPOSITION

Filed Dec. 29, 1945 2 Sheets-Sheet 1

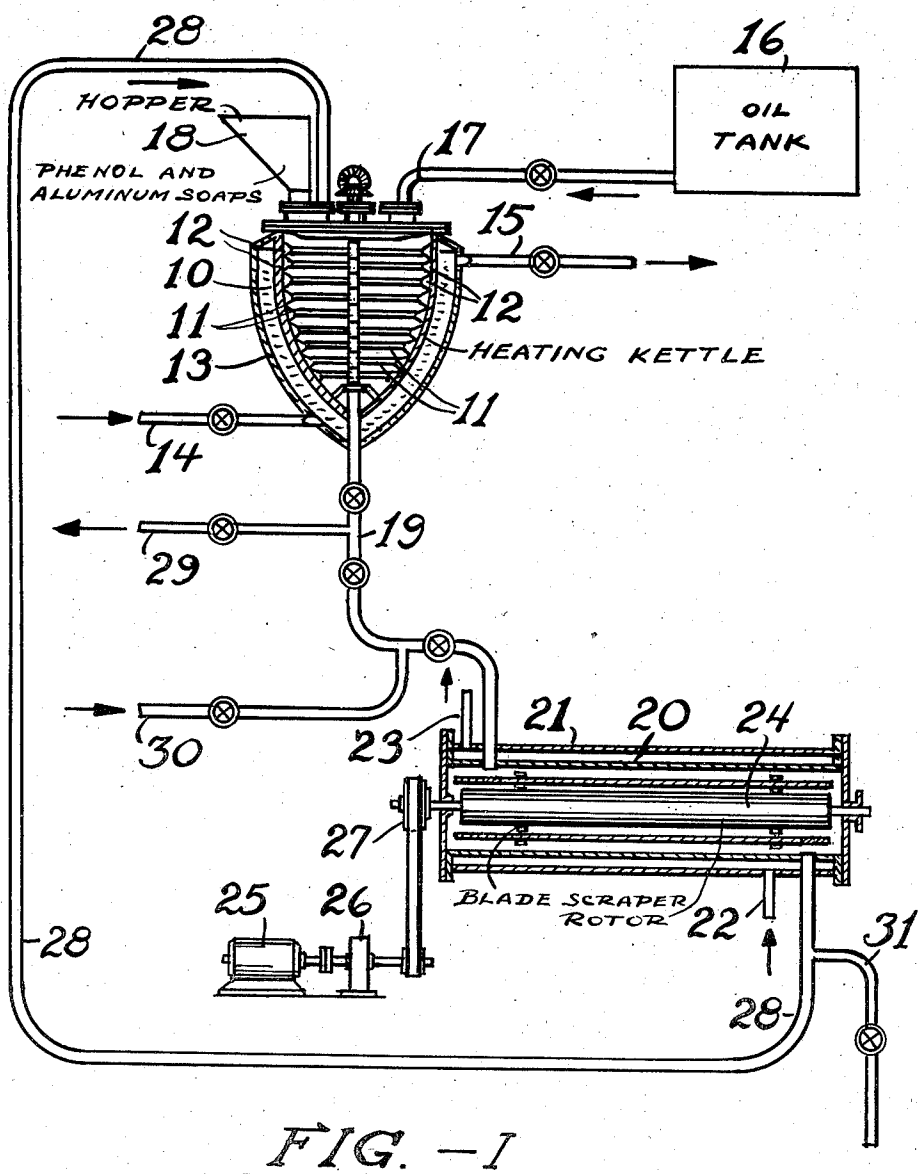


FIG. —1

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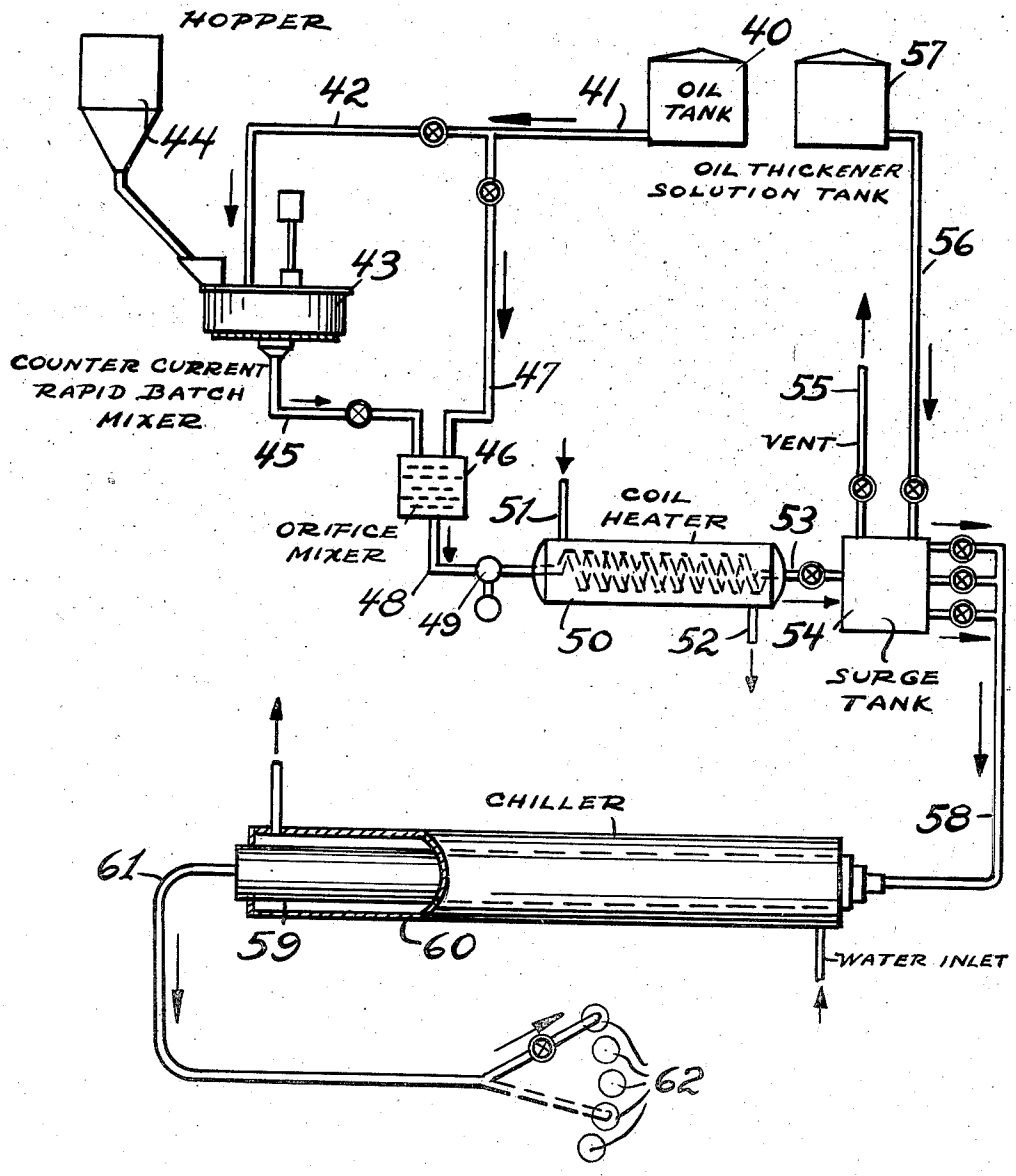


FIG.—II

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LUBRICATING GREASE COMPOSITION

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3 Claims. (Cl. 252—37)

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This invention relates to aluminum soap greases, and in particular to aluminum soap greases containing phenolic compounds.

In the lubricating industry, aluminum soap greases have a particularly important place as being outstandingly suitable for the lubrication of moving parts which require a measure of adhesiveness to the metal surfaces and good cohesion of the grease composition. Adhesiveness in a grease is important for the lubrication of parts such as the chassis of automobiles and crawler type tractors in which conditions prevail involving sudden shock causing splattering of the lubricant and exposure to a water-laden environment. Greases suitable for the lubrication of such parts are distinguishable from the strongly adhesive and coherent greases which are elastic in character and which show a greater tendency to separate from the metal parts and to ball up rather than to string out as required in such type lubrication. Aluminum soap greases are outstanding in their lubrication of chassis parts and for crawler type tractors. The present invention is concerned with an aluminum soap grease particularly suitable in the lubrication of such parts and in the use of a small amount of resorcinol as a crystallization modifier permitting the preparation of aluminum soap grease under more advantageous conditions than the prior art procedure of pan cooling.

The extensive use of aluminum soap greases has been somewhat curtailed by reason of difficulties of manufacture. The main difficulty in the preparation of aluminum soap greases is in regard to the obtaining of the desired homogeneous stringy structure. The desirable structure for the grease compositions has been obtainable in the past only by the laborious and slow pan cooling method. It has generally been considered that this slow cooling was necessary in order to avoid the crystallization of the aluminum stearate as the main soap constituent in the mineral oil after the high temperatures necessary for blending. For some time past, crystallization modifiers have been found which permit the rapid cooling of aluminum soap greases. The present invention relates to the use of resorcinol as a particularly advantageous crystallization modifier in the rapid cooling of aluminum soap greases from the high blending temperatures.

It has been found that when resorcinol in amounts between 0.075 and 3% is incorporated in the mineral oil-aluminum soap blends at temperatures of about 275–350° F., the composites can be cooled rapidly in bulk with agitation to

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temperatures as low as 85° F. and in layers of about ¼'' thick to temperatures as low as 50° F. without granulation or decomposition. This use of resorcinol 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. This advantageous effect of resorcinol has been found to occur, moreover, without the development of any disadvantageous effect upon the lubricating quality of the mineral oil-aluminum soap blend. In addition also, resorcinol in the grease compositions is not adversely affected by the incorporation of accessory additive materials such as oil thickening agents or anti-corrosive or anti-oxidation agents.

Thus, the present invention relates to the manufacture of highly stabilized aluminum soap grease compositions containing resorcinol as the special additive agent for a grease mixture of suitable mineral oil base and aluminum soap of a saturated fatty acid containing between 10 and 22 carbon atoms in the molecule, such as aluminum stearate. The amount in which the resorcinol is advantageously present to exert the crystallization inhibiting and stabilizing effect upon the final composition varies usually between 0.075% and 3% by weight of the total final composition. The preferable range of concentration of the resorcinol is between 0.1% and 1%. In such compositions, the aluminum soap is blended with the mineral oil to form a grease of suitable composition in amounts between 3% and 20%, but usually between 3% and 10%, by weight of the oil. Accessory ingredients to impart to the composites other desirable characteristics include free unsaturated fatty acids containing from 10 to 22 carbon atoms in the molecule and their aluminum soaps, naphthenic acids and their aluminum soaps and oil-thickening agents such as latex or polybutene.

The outstanding effect of the resorcinol appears to be connected primarily with the compounding and cooling steps in the preparation of the greases. It appears that the resorcinol functions in the aluminum soap-mineral oil blends to permit rapid cooling without crystallization of the aluminum soaps. Thus, the resorcinol permits the mixture after compounding to be agitated during cooling and the transition in structure to occur at lower temperatures than hithertofore in vessels of even 400 lbs. capacity. The resorcinol also exerts a definite but minor

stabilizing effect upon the cooled blended composition.

In preparing the grease compositions of this invention, the oil employed is preferably derived from a naphthene 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 resorcinol 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 resorcinol 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 this temperature until all of the soap and resorcinol 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 affected 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 scraped surface 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 the resorcinol, 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 scraped surface 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 drawing 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 330° 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 resorcinol supplied through opening **18** 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 scraped surface 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 scraper rotor **24** is operated by the motor **25** through gear box **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 of 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 setting 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 countercurrent rapid batch mixer **43**. To the mixer **43** is also added through the hopper **44** the required amounts of aluminum soap and resorcinol. The oil, the aluminum soaps and the resorcinol 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 **40** 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 **55** 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 scraped surface 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 examples are presented to illus-

trate the preparation and various characteristics of suitable greases prepared according to this invention:

Example 1

A grease of the following composition was prepared:

	Per cent
Resorcinol	0.29
Aluminum stearate	6.50
Polybutene solution (8% in oil of 200 S. U. vis./100° F.)	0.25
Lubricating oil of 70 S. U. vis./210° F	92.96

The aluminum stearate and one-sixth of the lubricating oil 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 remainder of lubricating oil, preheated to about 100° F. was added and agitation continued. The resorcinol and polybutene solution were then added and the temperature of the mixture raised to about 300° 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 77° F. of 275 and a worked penetration at 77° F. of 350.

Example 2

A grease of the following composition was prepared:

	Per cent
Resorcinol	0.27
Aluminum stearate	6.50
Lubricating oil of 70 S. U. vis./210° F	93.23

The materials were charged to a kettle as in Example 1, and heated to 310° F. The hot fluid grease was then pumped to a scraped surface chiller, 3.125 inches in inside diameter and 36 inches in inside length. The scraper consisted of two sets of blades, placed on opposite sides of the rotor, and hinged so as to be forced against the tube wall by their motion. The rotor was operated at 365 R. P. M. The grease was fed to the cooler at the rate of 2.48 pounds per minute, giving an outlet temperature of 96° F. (The water entered at 60° F. and left at 72° F.) The grease was drawn into containers and allowed to stand overnight, at which time it was found to be a clear, homogeneous plastic gel. Its unworked penetration at 77° F. was 279, and its worked penetration at 77° F. was 375.

Thus, it has been shown that satisfactory aluminum soap greases can be prepared in the presence of resorcinol 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 resorcinol obviates the time- and labor-consuming and the costly prior art process of preparing the 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 resorcinol 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 resorcinol 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 resorcinol 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 resorcinol, 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 is formed on the bearing surfaces a satisfactory adhesive lubricating film while the colloidal phase functions to offer substantial resistance 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 a lubricating grease which comprises thoroughly mixing at a temperature sufficiently high to cause the soap to dissolve in the oil, a viscous hydrocarbon oil, grease forming proportions of an aluminum soap of a saturated fatty acid containing between about 10 and 22 carbon atoms in the molecule, and 0.075 to 3% of resorcinol based on the total composition, rapidly cooling the heated mixture in a flowing stream by artificial chilling substantially below the normal transition temperature of about 140° F., and then allowing the mass to stand.

2. Process for preparing aluminum soap greases which comprises thoroughly mixing at a temperature sufficiently high to cause the soap to dissolve in the oil, a viscous mineral lubricating oil, about 3 to 10% of aluminum stearate and from about 0.1% to 1.0% of resorcinol based on the total composition, rapidly cooling the heated mixture by artificial chilling of a flowing stream of said mixture to a temperature substantially below normal transition range of about 140° F. and allowing the mass to stand.

3. 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 sufficiently high to cause the soap to dissolve in the oil, a viscous mineral lubricating oil of between 150–200 sec-

onds Saybolt at 210° F., about 3 to 10% of aluminum stearate and from 0.1% to 1.0% of resorcinol based on the total composition, rapidly cooling the heated mixture by artificial chilling in a flowing stream to a temperature of about 90° to 95° F., and then allowing the mixture to stand.

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JOHN C. ZIMMER.

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