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GREASE

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17 Claims. (Cl. 252—33.3)

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The present invention relates to block greases and more particularly relates to high temperature greases of the driving journal and rod cup types.

The so-called high temperature greases of the type of driving journal compound and rod cup greases are usually applied to the form of a cake or a block to the rotating shaft or bearing. Greases of these types are subjected to extreme pressure and temperature conditions, and therefore, must have high melting points in order to retain their shape and consistency at the elevated temperatures encountered in service; they must remain sufficiently plastic for extended periods of time in order to insure proper lubrication and at the same time they must be moldable or pressable in order to be fabricated in the desired form of cake or block, and also dispensable in pressure gun type lubricators. Greases of this type are soda soap greases containing from about 30% to about 60% soda soap of a fat or a mixture of a fat and fatty acid. An improvement in the preparation of greases of the herein described types was accomplished by incorporating in such greases a small amount of a preferentially oil-soluble sulfonic acid soap, as described in my Patent U. S. 2,352,811 of July 4, 1944. While greases prepared in accordance with teachings of this patent are satisfactory, it is necessary to use a fat, such as tallow or a mixture of fat and a fatty acid in making the soap stock. For various reasons, it is frequently advantageous to employ fatty acids as the sole soap stock instead of a fat or a mixture of a fat and fatty acids. Heretofore, this has not been practical since greases made solely with fatty acids possess undesirable characteristics and properties, such as crumbliness, oil leakage, and excessive loss of consistency on working or pressing.

It is an object of the present invention to provide a method of preparing high temperature soda soap greases having desirable pressing qualities. Another object of the invention is to provide an improved block grease made from fatty acids as the sole soap stock. Still another object of the invention is to provide a method of manufacturing a smooth short fiber high temperature soda soap grease from a fatty acid soap and a petroleum lubricating oil. Other objects and advantages of the invention will become apparent from the following description thereof.

Greases of the present invention comprise es-

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sentially an alkali metal soap of a long chain or high molecular weight fatty acid of at least about 12 carbon atoms, and preferably the soda soap of such fatty acids, an alkali metal salt, preferably the sodium salt, of a short chain organic acid of the classes described below, a petroleum oil, and an alkali metal soap of a preferentially oil-soluble sulfonic acid. For certain type of greases, particularly the so-called rod cup greases, it is desirable to incorporate a small amount, from about 0.1% to about 2%, of glycerine. The ranges for the various components of the greases are approximately as follows:

	Percent (by weight)
Alkali metal high molecular weight fatty acid soap	30 to 60
Alkali metal salt of a short chain aliphatic acid	0.1 to 10
Alkali metal oil-soluble sulfonate	1.0 to 20
Glycerine	0.0 to 2
Hydrocarbon oil	Remainder

The alkali metal salt of short chain organic acids suitable for the herein described invention are the salts of mono-basic saturated acids having the general formula RCOOH, or di-basic saturated acids having the general formula $R(COOH)_2$, or mixtures thereof, in which R is an aliphatic or an alkhydroxy group of not more than 6 carbon atoms. The organic radical R may be substituted with hydrocarbon and/or non-hydrocarbon groups, such as halogen, amines and the like. Specific examples of suitable organic acids are formic acid, acetic acid, hydroxyacetic acid, lactic acid, propionic acid, butyric acid, iso-butyric acid, valeric acid, trimethyl acetic acid, caproic acid, hydroxycaproic acid, oxalic acid, malonic acid, succinic acid, methyl malonic acid, tartaric acid, dihydroxy maleic acid and the like. While each of the foregoing specific acids is suitable for use in the present invention, the improvement obtained and the degree of improvement affected may vary to some extent with the acid used.

Preferentially oil-soluble sulfonic acids employed in accordance with the present invention are preferably those obtained in the treatment of hydrocarbon oils such as mineral or petroleum oils with strong acid, i. e., concentrated or fuming sulfuric acid. However, for the purpose herein described we can use other preferentially oil-

soluble sulfonic acids, such as those obtained by the sulfonation of olefin polymers having at least about 22 carbon atoms in the molecule or those obtained by the sulfonation of alkylated aromatics having at least about 19 carbon atoms in the alkyl chain.

The preferentially oil-soluble petroleum sulfonates are preferably those obtained in the treatment of petroleum oils to obtain highly refined products of the type of electrical insulating oils, turbine oils, medicinal white oils, technical white oils, etc., in which the petroleum oils are treated successively with a number of portions of concentrated sulfuric acid (i. e., above about 95% strength), or fuming sulfuric acid. A variety of sulfur-containing compounds are formed by the chemical reactions of sulfuric acid upon the oil, including sulfonic acids, organic esters of sulfuric acid, partial esters of sulfuric acid, etc. Most of these compounds are relatively insoluble in the oil under the treating conditions and separate from the oil together with unreacted sulfuric acid as a sludge, which is separated from the oil after each treatment. The sulfuric acid is usually added in "dumps" of about one-half pound per gallon of the oil, the total quantity of acid added depending upon the oil being treated and the desired final product. Usually from about three pounds to about nine pounds of sulfuric acid per gallon of oil is used. Some of the sulfonic acids resulting from the treatment of the oil with the sulfuric acid are preferentially oil-soluble and remain in the oil layer after removal of the acid sludge. These can be removed from the oil by neutralizing the acid-treated oil with an alkaline agent, such as ammonia, or an alkali metal hydroxide, preferably sodium hydroxide to form sulfonic acid soaps or sulfonates which are then extracted from the oil by treatment with 50% to 80% aqueous alcohol solutions or other suitable means. Because of the characteristic mahogany color of these sulfonates they are known in the petroleum art as mahogany soaps. While the majority proportions of the preferentially oil-soluble sulfonates are obtained from the acid-treated oil there can be recovered from the acid sludge, by suitable solvents, preferentially oil-soluble sulfonates or sulfonic acids. The term "preferentially oil-soluble" sulfonates therefore includes the oil-soluble sulfonates obtained from both the acid-treated oil and the acid sludge.

While any of the preferentially oil-soluble sulfonates can be used, I prefer to employ those obtained from oil-soluble sulfonic acids having combined weights within the range of from about 350 to about 525, and particularly in the range of about 450 to about 500. The combining weights of the oil-soluble petroleum sulfonic acids vary with the viscosity of the oil being acid-treated and the total amount of sulfuric acid employed. To a certain degree the type of preferentially oil-soluble petroleum sulfonic acid obtained will also depend upon the type of crude oil from which the acid-treated oil is obtained. For example, the preferentially oil-soluble sulfonic acids obtained in treating a petroleum distillate having a Saybolt Universal viscosity at 100° F. of from about 60 seconds to about 230 seconds with 3 to 5 pounds of fuming sulfuric acid, have combining weights of about 430, while the preferentially oil-soluble sulfonic acids obtained when treating petroleum distillates having Saybolt Universal viscosities at 100° F. of from about 220 seconds to about 800 seconds with from about three to about nine pounds of fuming sulfuric acid per gallon of oil have combining weights of from about 470 to about 500.

Generally, we prefer to use preferentially oil-soluble petroleum sulfonates of the metals of group I of the periodic system, particularly sodium and potassium salts, although we may use sulfonates of metals or other groups of the periodic system or of ammonium or ammonia derivatives, such as amines and the like.

While as stated above, we can use metal salts of preferentially oil-soluble petroleum sulfonic acids having combining weights within the range of from about 350 to about 525, we prefer to use the metal salts of petroleum sulfonic acids having combining weights within the range of from about 450 to about 500. Specifically, we have obtained excellent results with sulfonates of the type obtainable by treating petroleum distillates of from about 200 seconds to about 800 seconds Saybolt Universal viscosity at 100° F., with from about three to about nine pounds of strong sulfuric acid, preferably fuming sulfuric acid, per gallon of oil. The method of obtaining desirable soaps of preferentially oil-soluble sulfonic acids derived from petroleum oils is illustrated by the following examples which describe the preparation of a sodium soap of the preferentially oil-soluble sulfonic acids having combining weights of about 470 to about 500.

A petroleum oil distillate having a Saybolt Universal viscosity at 100° F. of about 650 seconds is treated with from about 3 to about 6 pounds of fuming sulfuric acid per gallon of oil in one-half pound increments or "dumps." After the acid sludge from each one-half pound acid "dump" is settled and withdrawn, the next one-half pound of fuming sulfuric acid is added to the oil. The temperature of the oil before the fuming acid is added thereto is maintained below at about 60° F. but due to the heat of reaction upon addition of the sulfuric acid, the temperature of the oil may rise from about 90° F. to about 100° F. After the required total amount of fuming sulfuric acid has been added to the oil and the oil freed of acid sludge, the acid-treated oil containing oil-soluble sulfonic acids dissolved therein is neutralized with a solution of sodium hydroxide. The aqueous alkali solution is then separated from the oil solution containing dissolved therein sodium soaps of sulfonic acids and the latter separated from the oil by extraction with alcohol of about 60% strength. The alcohol layer containing dissolved sodium sulfonates is then separated from the oil and subsequently distilled to recover the alcohol and remove water. The crude sulfonic soap obtained in this manner contains from about 30% to about 60% sodium sulfonate, from about 30% to about 60% oil, from about 1% to about 10% water, and up to 10% inorganic salts, which may be removed by the procedure hereinafter described.

The above procedure may be modified in that after the acid sludge is removed from the acid-treated oil, the oil containing dissolved sulfonic acids is extracted with about 60% alcohol to remove the sulfonic acids which may then be neutralized with sodium hydroxide and subsequently freed of the alcohol by distillation.

A preferentially oil-soluble petroleum sulfonate of similar solubility characteristics can be obtained by similarly treating a Mid-Continent crude distillate oil having a viscosity of about 350 seconds Saybolt Universal at 100° F. with a total of five pounds of fuming sulfuric acid.

Soaps of preferentially oil-soluble sulfonic acids having similar combining weights and properties are obtainable by treating a distillate having a Saybolt Universal viscosity at 100° F. of from about 500 to about 850 seconds with about 6 to 9 pounds of fuming sulfuric acid per gallon of oil.

The crude soaps of these preferentially oil-soluble sulfonic acids obtained by the procedure described above may be freed of inorganic salts by purification. This purification is preferably accomplished by dilution of the crude soap with from about ½ to about 10 parts, preferably 1 to 2 parts of 50% or higher strength alcohol, preferably alcohol of 60% to 70% strength, and allowing the salts to settle while maintaining the mixture within the temperature range of 130° F. to 175° F., preferably 155° F. to 165° F. When the salts have settled, the supernatant alcohol-soap layer is separated and the alcohol is recovered by conventional distillation procedure. By this method of purification the salt content of the crude sulfonic soap can be readily reduced to 5% or less, e. g., to about 3.5%.

The long chain fatty acids employed in preparing the soap stock can be unsaturated, partially unsaturated or saturated fatty acids, having from 12 to 22 or more carbon atoms in the chain, such as for example, lauric acid, palmitic acid, stearic acid, oleic, vegetable fatty acid, such as for example, cottonseed fatty acid, animal fatty acids, standard fatty acids, behenic acid and hydrogenated fish fatty acids or the fatty acid pitch obtained therefrom in accordance with the procedure demonstrated in U. S. Letters Patent Nos. 2,229,367 and 2,229,368. The standard fatty acid is a product well known in the grease-making trade and comprises a mixture of about 40% animal fatty acids and about 60% cottonseed fatty acids.

The hydrocarbon oil constitutent can be a synthetic or a natural hydrocarbon oil, such as a petroleum oil in the viscosity range of from about 80 seconds at 100° F. to about 300 seconds at 210° F. Saybolt Universal viscosity, the viscosity selected being dependent upon the intended use or service for the grease.

The greases of the present invention can be made at fire kettle temperatures, namely above 320° F. or by the steam kettle method at temperatures of 250–320° F., although the present invention is particularly adaptable to the preparation of fire kettle greases. In preparing the greases by the fire kettle method, all of the fatty acid, the short chain organic acid, the oil-soluble sulfonate, and a small amount of the hydrocarbon oil are charged to the mixture and heated to about 200–210° F. The alkali metal saponifying or neutralizing agent, such as caustic soda in a 48° Baumé solution, is then added to the mixture and the mass gently heated with stirring until substantially dry, that is, practically all of the water has been evaporated from the mixture. The temperature of the batch is then raised to a temperature of from about 320° F. to about 380° F., and preferably to about 350° F., the balance of the hydrocarbon oil added, and the alkalinity adjusted to give a product with about 0.2% excess alkalinity, e. g., 0.2% sodium hydroxide. The batch is then heated from about 530° F. to about 570° F., and preferably to about 550° F., and maintained at this temperature until clear and free of foam. Heating of the batch is then discontinued and the material cooled to a temperature not below about 300° F., and preferably to a temperature of 510° F. to about 490° F. for filling.

Optionally, instead of forming in situ the alkali metal salt of the short chain fatty acid, an alkali metal salt of such acids may be used.

In preparing greases suitable as rod cup lubricants, which are usually dispensed through pressure gun-type lubricators, it is desirable to incorporate a small amount, preferably from about 0.1% to about 2% of glycerine in the grease. The preparation of such grease differs somewhat from the above-described method in that the procedure above-described is followed to the point at which the grease is cooled to a temperature of about 490° F. to about 510° F., at which time the glycerine is added. As soon as the foaming, which usually takes place at this time has subsided, and the batch is clear, it is ready for filling. If desired, a small amount, namely from about 0.001% to about 0.0001% of a silicone polymer may be added to inhibit foaming. In case a grease having longer fibers is desired, the grease is cooled to a temperature as low as 350° F. or 300° F., before it is filled.

Greases made in accordance with the hereindescribed invention have improved penetration and softening characteristics. Penetration is an indication of hardness and consistency and in the following data it has been based on the A. S. T. M. penetration data test, D217–38T. Softening time, on the other hand, is an index of quality from the service standpoint and is measured by the following described test. A cylinder of the grease, one-half inch in diameter and about five-eighths inch long is heated on a metal plate contained in an oil or mercury bath. The temperature of the bath is controlled to give the desired temperature, usually about 340° F., at point of contact between the plate and the grease sample which is subjected to the weight of an 85 gram cylinder, thus providing a pressure on the driving journal compound comparable to the pressure to which the same is subjected in actual operation when it is forced by a spring against a hot bearing. Softening time is defined as the time in seconds required to flatten the test cylinder one-fourth inch under these conditions. If the test sample does not flatten one-fourth inch at the end of 300 seconds, the test is terminated and the amount the test sample has flattened noted; the result is expressed thus, for example "300 (⅛)", which means at the end of 300 seconds the test piece flattened one-eighth of the total movable distance of ¼ inch. High temperature greases made from fatty acid by the prior art methods usually have a softening time varying from between about 80 seconds and 200 seconds.

The advantages of the present invention are demonstrated by the following examples, which are given by way of illustration only and not intended to limit the scope of the invention.

Example	1	2	3
Percent Soda Tallow fatty acid soap	47.5	47.5	47.5
Percent Sodium formate	1.0	3.0	0.0
Percent Sodium Mahogany Soap	6.0	6.0	6.0
Percent Petroleum Oil (S. U. Vis. at 210° F.=200–210 seconds)	45.5	43.5	46.5
Penetration at 77° F	29	25	53
Softening time at 340° F. (85 g. wt.)	300(⅛)	300(⅛)	162 secs.
Structure	Good	Good	
Pressability	Good	Good	(1)

[1] Grease is sticky on working; not pressable.

The grease of Example 3, made as the greases

of Examples 1 and 2, but without the short chain organic acid salt is inferior to the greases of Examples 1 and 2 in that it became sticky on being worked and is not pressable.

Example	4	5	6
Percent Soda tallow fatty acid soap	47.5	47.5	47.5
Percent Sodium lactate	1.0	4.0	4.0
Percent Sodium Mahogany soap	6.0	6.0	0.0
Percent Petroleum Oil (S. U. Vis. at 210° F.=200-210 secs.)	45.5	42.5	48.5
Penetration at 77° F	34	30	45
Softening time at 340° F. (85g. wt.)	300(⅛)	300 (no drop)	300(⅛)
Structure	Good	Excellent	Good
Pressability	Good	Excellent	(1)

[1] Not pressable; sticky on working.

The advantage of using a soap of an oil-soluble sulfonic acid in combination with the salt of a short chain organic acid is demonstrated by the above examples. Example 6 was prepared exactly as were Examples 4 and 5 but without the sodium mahogany soap; however, whereas the greases of Examples 4 and 5 were pressable, the grease of Example 6 became sticky on being worked and was not pressable.

Example	7	8	9
Percent Soda Tallow fatty acid soap	47.5	47.5	45.3
Percent Sodium hydroxy acetate	1.3	6.5	0.9
Percent Sodium formate	0.0	0.0	1.0
Percent Sodium mahogany soap	5.0	5.0	5.0
Percent Petroleum oil (S. U. Vis. at 210° F.=200-210 secs.)	46.2	41.0	47.8
Penetration at 77° F	32	34	32
Softening time at 340° F. (85 g. wt.)	300(⅞)	300(⅞)	300(¼)
Structure	Fair	Very good	Good
Pressability	Fair	Very good	Very good

Example 9 demonstrates the practicability of preparing satisfactory greases with a combination of salts of different short chain organic acids.

Example	10	11
Percent Soda tallow fatty acid soap	48.0	48.0
Percent Sodium formate	0.0	0.5
Percent Sodium mahogany soap	5.0	5.0
Percent Glycerine	0.2	0.2
Percent Petroleum oil (S. U. Vis. at 210° F.=200-210 secs.)	46.8	46.3
Penetration at 77° F	30	26
Softening Time at 340° F. (85 g. wt.)	300(1/16)	300 (no drop)
Structure	(1)	Very good
Pressability	(2)	Very good

[1] Fair, slightly crumbly.
[2] Fair, slightly sticky.

Example 11 represents a composition suitable for rod-cup greases. A small amount of glycerine is desirable in this type of grease to facilitate its dispensability at low temperatures. The grease composition represented by Example 10 is similar to that of Example 11 except that the former does not contain sodium formate; resulting in a grease slightly inferior to the grease of Example 11 for its intended use. The grease of Example 10 was not as good as the grease of Example 11 as a rod-cup lubricant in that the former did not dispense as readily as the latter in a Prime Rod Lubricator at temperatures of about 20° F.

Greases made in accordance with the present invention may contain small amounts of ingredients other than the essential components named herein. For example, there may be incorporated in such greases small amounts of an anti-oxidant such as a naphthol, phenyl alpha naphthylamine, etc., extreme pressure agents, such as halogenated hydrocarbons, mica, graphite, etc.

Percentages of greases herein and in the appended claims are weight percentages.

While I have described my invention with particular reference to certain preferred embodiments thereof, it is to be understood that these are by way of illustration and not by way of limitation.

I claim:

1. A high temperature grease comprising from about 30% to about 60% of an alkali metal soap of long chain fatty acids of at least about 12 carbon atoms, from about 1% to about 20% of an alkali metal soap of a preferentially oil-soluble sulfonic acid, from about 0.1% to about 10% of an alkali metal salt of a short chain aliphatic acid having not more than 6 carbon atoms in the chain, and a hydrocarbon oil.

2. A high temperature grease comprising from about 30% to about 60% of an alkali metal soap of a long chain fatty acid of at least about 12 carbon atoms, from about 0.1% to about 10% of an alkali metal salt of a short chain organic acid selected from the group consisting of a mono-basic saturated acid having the general formula RCOOH and a di-basic saturated acid having the general formula $R(COOH)_2$, and mixtures thereof, in which R is an organic group selected from the class consisting of an aliphatic group containing not more than 6 carbon atoms and an alkhydroxy group containing not more than 6 carbon atoms, irom about 1% to about 20% of an alkali metal soap of a preferentially oil-soluble sulfonic acid and the remainder hydrocarbon oil.

3. A high temperature grease as described in claim 2 in which the alkali metal is sodium.

4. A high temperature grease as described in claim 2 in which the high molecular weight fatty acid is tallow fatty acids.

5. A high temperature grease as described in claim 2 in which the high molecular weight fatty acid is stearic acid.

6. A high temperature grease as described in claim 2 in which the high molecular weight fatty acid is a mixture of animal fatty acids and vegetable fatty acids.

7. A high temperature grease as described in claim 2 in which the short chain aliphatic acid is formic acid.

8. A high temperature grease as described in claim 2 in which the alkali metal aliphatic acid is a lactate.

9. A high temperature grease as described in claim 2 in which the short chain aliphatic acid is hydroxy acetic acid.

10. A high temperature grease of the rod cup-type comprising from about 30% to about 60% of a soda soap of a high molecular weight fatty acid of at least about 12 carbon atoms, from about 0.1% to about 10% of a sodium salt of a short chain organic acid selected from the class consisting of a mono-basic saturated acid having the general formula RCOOH, a di-basic saturated acid having the general formula $R(COOH)_2$ and mixtures thereof, in which R is an organic group selected from the class consisting of an aliphatic group of not more than about 6 carbon atoms, and an alkhydroxy group of not more than about 6 carbon atoms, from about 1% to about 20% of the sodium soap of a preferentially oil-soluble sulfonic acid, from about 0.1% to about 2% glycerine and the remainder a hydrocarbon oil.

11. A high temperature grease as described in

claim 10, in which the high molecular weight fatty acid is tallow fatty acid.

12. A high temperature grease as described in claim 10 in which the short chain aliphatic acid is formic acid.

13. A high temperature grease as described in claim 10 in which the short chain aliphatic acid is hydroxyacetic acid.

14. A high temperature grease comprising about 48% soda soap of a high molecular weight fatty acid having at least 12 carbon atoms in the molecule, about 5% of sodium soap of preferentially oil-soluble sulfonate, about 0.5% sodium formate, about 0.2% glycerine, and the remainder petroleum oil.

15. The method of preparing a high temperature grease comprising from about 30% to about 60% of an alkali metal soap of high molecular weight fatty acids, from about 0.1% to about 10% of an alkali metal salt of a short chain organic acid selected from the group consisting of a monobasic saturated acid having the general formula RCOOH, a di-basic saturated acid having the general formula $R(COOH)_2$, and mixtures thereof, in which R is an organic group selected from the class consisting of an aliphatic group containing not more than about 6 carbon atoms and an alkhydroxy group containing not more than about 6 carbon atoms and about 1% to about 20% of an alkali metal soap of a preferentially oil-soluble sulfonic acid and the remainder a hydrocarbon oil comprising the following steps: mixing all of the fatty acid, the short chain organic acid, the oil-soluble sulfonates, and a small amount of the hydrocarbon oil in a mixer and heating the same to about 200–210° F., adding an aqueous solution of the alkali metal saponifying agent and stirring the mixture until substantially all of the water is evaporated therefrom, raising the temperature of the mixture from about 320° F. to about 380° F., adjusting the alkalinity of the mixture to about an excess of 0.2% alkali, heating the alkalinity-adjusted mixture at a temperature of from about 530° F. to 570° F. until the mixture is clear and free of foam, cooling the mixture to a temperature not below about 30° F. and filling the same.

16. The method of preparing a high temperature grease comprising from about 30% to about 60% of an alkali metal soap of high molecular weight fatty acids, from about 0.1% to about 10% of an alkali metal salt of a short chain organic acid selected from the group consisting of a monobasic saturated acid having the general formula RCOOH, a di-basic saturated acid having the general formula $R(COOH)_2$ and mixtures thereof, in which R is an organic group selected from the class consisting of an aliphatic group containing not more than about 6 carbon atoms and an alkhydroxy group containing not more than about 6 carbon atoms, from about 1% to about 20% of an alkali metal soap of a preferentially oil-soluble sulfonic acid, from about 0.1% to about 2% glycerine, and the remainder a hydrocarbon oil comprising the following steps: mixing all of the fatty acid, the short chain organic acids, the oil-soluble sulfonates, and a small amount of the hydrocarbon oil in a mixer and heating the same to about 200–210° F., adding an aqueous solution of the alkali metal saponifying agent and stirring the mixture until substantially all of the water is evaporated therefrom, raising the temperature of the mixture to about 320° F. to about 380° F., adjusting the alkalinity of the mixture to about an excess of about 0.2% alkali, heating the alkalinity-adjusted mixture at a temperature of from about 530° F. to about 570° F. until the mixture is clear and free of foam, cooling the mixture to a temperature of 490-510° F., adding from about 0.1% to about 2% glycerine, and filling the grease when substantially all foaming has subsided.

17. The method of preparing a high temperature grease as described in claim 16, in which the grease is cooled and filled at a temperature below about 490° F. and above about 300° F., after the addition of glycerine.

REUBEN A. SWENSON.

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The following references are of record in the file of this patent:

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Certificate of Correction

Patent No. 2,487,080 November 8, 1949

REUBEN A. SWENSON

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows:

Column 9, line 46, for "about 30° F." read *about 300° F.*;

and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 21st day of March, A. D. 1950.

[SEAL]

THOMAS F. MURPHY,
Assistant Commissioner of Patents.