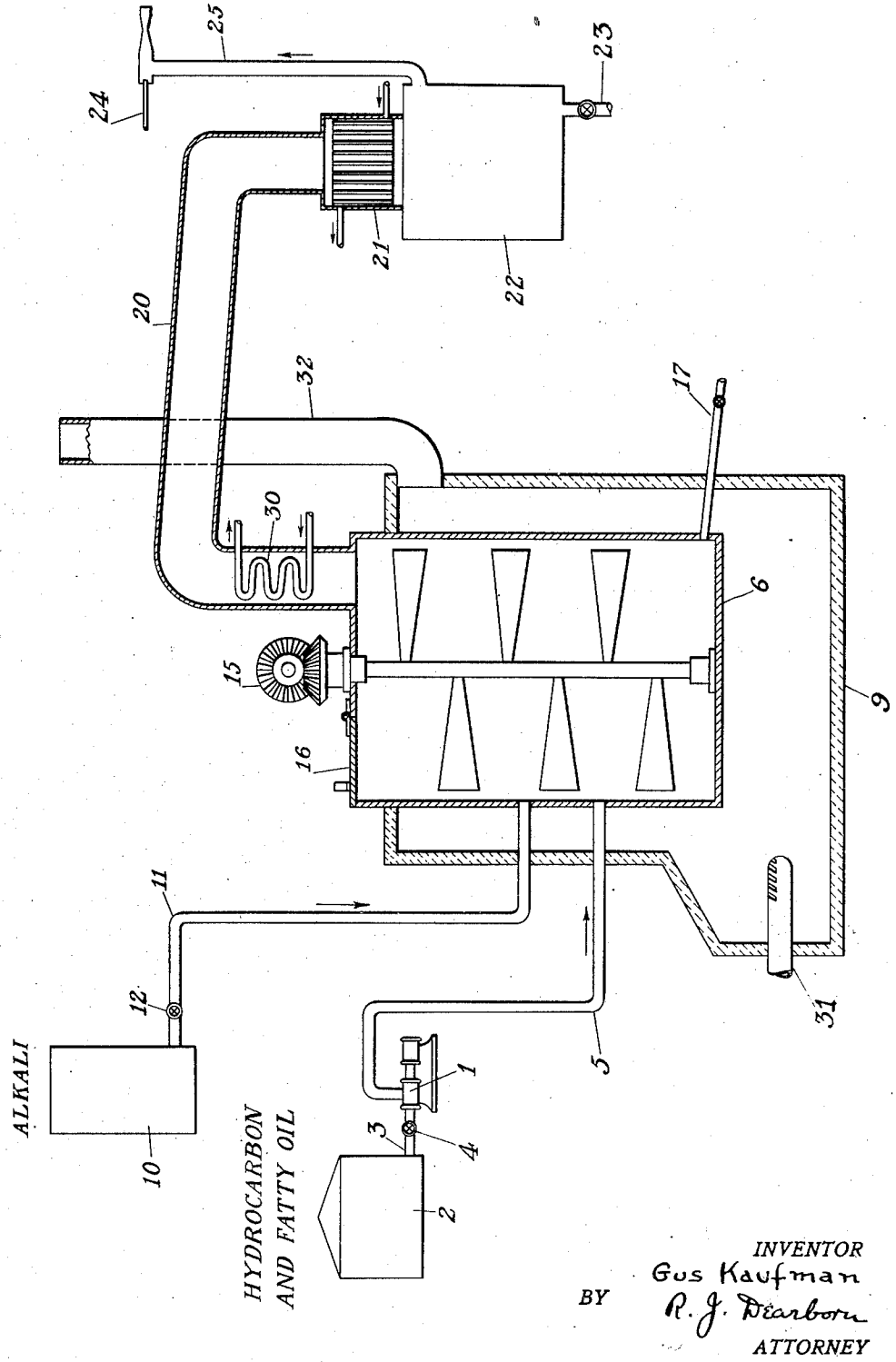
Aug. 28, 1934. G. KAUFMAN 1,971,750

GREASE AND ITS MANUFACTURE

Filed Aug. 17, 1931



INVENTOR
Gus Kaufman

BY R. J. Dearborn
ATTORNEY

Patented Aug. 28, 1934 1,971,750

UNITED STATES PATENT OFFICE

1,971,750

GREASE AND ITS MANUFACTURE

Gus Kaufman, Beacon, N. Y., assignor to The Texas Company, New York, N. Y., a corporation of Delaware

Application August 17, 1931, Serial No. 557,643

2 Claims. (Cl. 87—9)

This application is a continuation-in-part of my copending application filed July 1, 1930, Serial No. 465,145.

This invention comprises a method of heat treating oils used in the preparation of lubricating greases, and particularly heat treating a mixture of a mineral oil and a soap forming material, such as a fatty oil, fatty acid and the like, and also the products resulting from such treatment; following the heat treatment the mixed oils may be subjected to saponification to produce the desired lubricating grease. The invention further comprises the controlled dephlegmation of vapors arising in such heat treating and saponification or in either of these steps.

Broadly, my invention contemplates the preparation of grease or lubricating compositions composed mainly of hydrocarbon oil and a soap prepared and combined in such a manner that the physical properties of the resulting grease, such as melting point and hardness, may be varied to a considerable extent substantially independently of the soap content.

The invention contemplates an improved method of manufacture of grease compositions wherein the hydrocarbon oil and fatty oil ingredients are preliminarily heated, preferably in admixture, under various conditions of temperature or during various periods of time at various temperatures in order to effect a digestion or conversion. The extent of this conversion may conveniently be controlled by regulating the increase in viscosity to a predetermined point. When the desired extent of conversion has been effected, akaline material is added to react with the saponifiable constituents of the digested oil mixture to form a final product of desired physical and chemical characteristics.

It has been common in the prior grease making art to heat the mineral oil or hydrocarbon and fat or fatty acid together to the desired temperature for carrying out saponification, which might have been in some instances as high as 400 to 500° F. The aqueous solution of saponifying agent was then added without further delay in such regulated quantities that the water would be flashed off continuously, thus avoiding the possibilities of boil-overs due to excesive foaming. The time of manufacture of a batch was also materially reduced by this method.

According to the present invention the mineral oil and fat or fatty acid are not only heated together to a high temperature, but they are maintained at a high temperature for a substantial period during which the aforementioned increase in viscosity occurs. In the light of the prior art methods this heating may be said to be "needlessly" prolonged.

The physical and chemical properties of greases made according to the methods of the prior art are largely dependent upon the nature of the ingredients used in their preparation. Thus it has been customary, for example, to control the hardness of grease by varying the soap content, the hardness of the grease as commonly prepared usually being roughly directly proportional to its soap content.

Even when very high melting point or high titer fats are employed in the preparation of a hard grease by the prior art methods, the soap content of the resulting product may very greatly exceed that of my product having the same hardness and prepared from lower melting point fats.

By following the method of my invention an improved grease is produced, certain of the characteristics of which may be varied at will, but generally speaking one which has a melting point appreciably lower than that of grease of corresponding hardness manufactured by conventional methods of practice from substantially similar type ingredients. Greases made by employing my invention may contain materially lower proportions of soap than greases prepared by the prior art methods. In other words, if it is desired to produce a grease having a certain consistency or hardness the soap content necessary to attain such consistency or hardness may be reduced to any desired extent up to 30 to 50% less soap and in some instances considerably greater reduction by the use of my invention. As a consequence of this the resultant grease will contain a larger proportion of hydrocarbon or lubricating oil, thus tending not only to very materially increase the lubricating qualities but also to greatly reduce the cost, inasmuch as a portion of the more expensive soap ingredient will have been replaced by the less expensive mineral oil ingredient.

Likewise it has been found that lower viscosity lubricating oils may partially or wholly replace higher viscosity lubricating oils in the preparation of greases by my method, while attaining the same consistency or hardness as when using the more viscous oils. By the substitution of lower viscosity oils for higher viscosity oils while maintaining a given consistency or hardness, considerable advantages have been experienced in reducing the frictional resistance on account of the presence of the lower viscosity lubricating oil.

Another aspect of the invention contemplates subjecting the vapors evolved during manufac-

ture of the grease to controlled dephlegmation whereby certain valuable constituents of the vapors may be recovered and either returned to the mixture undergoing treatment or may suitably be disposed of in the preparation of other grease compositions, as well as improved apparatus adapted to carry out the invention.

Broadly speaking, my invention contemplates the preparation of lubricating compositions or greases, having desired lubricating and other physical properties, which may be composed of mineral and fatty oil or fatty acid ingredients of varying nature and physical characteristics and wherein the physical properties of the final product, particularly its consistency or hardness, may substantially be controlled irrespective of either its soap content or of the particular type of mineral or fatty oil used in its preparation. The petroleum or hydrocarbon lubricating oil constituents may be of the nature of a cylinder oil or cylinder stock and may embrace either a distillate or a residual product derived from either paraffin or naphthene base, or mixed paraffin and naphthene base crudes, or, on the other hand, may be of the nature of a pale oil or a fraction of lower boiling range than cylinder oil though derived from any of the foregoing crude sources. The fatty oil or fatty acid may be derived from either animal or vegetable origin, such as hard tallow, tallow oil, fleshing grease, stearin, elaine oil, castor oil, cottonseed oil, residual pitchy material resulting from the destructive distillation of fatty oils and fatty acids, and the like, and may also embrace hydrogenated fats.

The general procedure followed in preparing lubricating compositions in accordance with my invention is as follows: The hydrocarbon or mineral lubricating oil and fatty oil ingredients are mixed in suitable proportion, depending upon the quantity of these ingredients desired in the final product and are heated to a desired digestion or conversion temperature. During this heat treatment the oils are preferably continuously stirred. This temperature of treatment may range from approximately the melting point of the resulting grease, as for example 400° F., or even lower, to upwards of 600° F. The heated mixture of oils is then maintained substantially constant at the desired temperature for a predetermined period of time or until the mixture attains a desired degree of digestion or conversion, preferably determined by a progressive increase in the viscosity of the admixture. The digested mixture is then saponified in the usual manner by addition thereto of a suitable alkaline material such as caustic soda, lime, or the like, either with or without prior cooling of the digested mixture.

Although it is usually preferable to subject the petroleum and fatty oil ingredients to heat treatment after mixing, it is contemplated that these ingredients may be separately subjected to heating or digestion with subsequent mixing while in a heated state prior to saponification for the production of certain lubricating compositions.

During digestion or heat treatment the mineral and fatty oils may undergo partial decomposition or conversion with the formation of certain reaction products which remain in the mixture as well as more volatile fractions, which latter may include glycerine, fatty acids such as oleic, stearic, palmitic, etc. and hydrocarbon fractions. These evolved substances in the form of a vapor are preferably subjected to controlled dephlegmation for the removal of the valuable fatty acid and other constituents to form a reflux condensate which may be returned to the mixture undergoing treatment.

Although this reflux condensate is preferably returned to the chamber or kettle, it is also contemplated withdrawing it directly from the dephlegmating means for disposition in some other desirable manner, for example, as a fatty oil ingredient in the preparation of certain greases or lubricating compositions for which it may be particularly well adapted.

While the exact nature of the physical and chemical changes occurring during digestion of the mixture has not been determined, apparently certain new compounds or products of reaction are formed which impart desirable qualities, especially that of hardness, to the resulting final product and which may be formed as the result of reaction during digestion or conversion, between the mineral and fatty oil, between constituents thereof, or between compounds or products formed as a result of conversion or partial decomposition of the fatty and mineral oils during the heat treatment or as the result of a combination of such reactions. These reactions may result in part at least from oxidation of the hot mixture resulting from the contact of the atmosphere with the surface of the mixture, or the mechanical stirring into the mixture of some of the air over it. This theory is strengthened by reason of the fact that it has been found that the desired increase in viscosity, and resulting increase in the hardness of the final grease, is hastened somewhat by the introduction of air into the mixture.

These reactions are usually accompanied by corresponding and progressive changes in the physical characteristics of the mixture during digestion, such as discoloration, and increasing viscosity so that the duration of heat treatment may readily be controlled by observation of these progressive physical changes and is preferably determined by the change in viscosity of the digesting mixture.

The invention may further be described in reference to the accompanying drawing illustrating diagrammatically a preferred embodiment of the apparatus adapted to practice the invention.

In the drawing, a pump 1 withdraws hydrocarbon and fatty oil which has been previously mixed in proper proportion in a tank 2 through a pipe 3 having a valve 4 and delivers it through a pipe 5 to a kettle or chamber 6 wherein the mixture is subjected to heat treatment with subsequent saponification of its saponifiable matter.

The kettle is preferably placed within a furnace 9 the upper walls of which enclose substantially all of the kettle leaving only a relatively small upper portion exposed including the top thereof projecting above the furnace walls. The latter are suitably spaced from the shell of the kettle to form an annular space in which hot combustion gases may surround the kettle as they rise through this annular chamber on their way to the stack. The kettle may be equipped with conventional stirring paddles or like mechanism driven through suitable bevel gears 15 operated by a source of power, not shown.

Alkali from tank 10 may be withdrawn through pipe 11 having a valve 12 and introduced into the kettle 6. The kettle 6 may also be provided with a hinged cover 16 and a draw-off pipe and valve 17. A pipe 20 adapted to withdraw the fumes rising in the kettle 6 is connected to a condenser 21, preferably water-cooled, and condensate which may be condensed in the con-

denser 21 flows into receiver 22, provided with a draw-off pipe and valve 23. In order to promote the exhaust of fumes from the kettle 6 through the pipe 20 and into condenser 21 and receiver 22, a steam jet 24 is connected by a pipe 25 to the upper part of receiver 22. The pipe 20 is also preferably provided with a cooling coil 30 through which regulated quantities of a cooling medium may be passed. The furnace 9 is provided with the customary burner 31 and waste flue gases are permitted to pass from the upper part of the furnace to the stack 32.

The oil mixture is preferably heated to the desired digestion or reaction temperature as rapidly as possible after which it may be maintained substantially constant at the desired temperature by controlling the rate of firing the furnace until the mixture has attained a predetermined viscosity. Thus the mixture may be raised to a relatively low temperature, for example from 400° F. to 480° F., and be maintained at substantially this temperature for a relatively long period of time, while on the other hand the oil may be heated to a relatively higher temperature, ranging from 500° F. to 700° F. or even higher, at which temperature it may be maintained for a relatively short period of time in order to bring about the desired degree of conversion.

When heating or digestion is completed, saponification may be brought about by the addition of caustic soda or other suitable soap forming chemicals to the digested or reacted mixture, preferably without prior cooling of the mixture. On the other hand, cooling may be desirable prior to the addition of the alkaline or basic material depending upon the temperature of the heat reacted mixture, in order to prevent excessive foaming or frothing during its addition.

The saponifying agent is withdrawn from the tank 10 through the pipe 11 into the kettle 6 and is preferably added very slowly with continued agitation of the contents of the kettle by the paddles in order to avoid excessive frothing or foaming.

Upon completion of saponification the resulting grease preferably is drawn from the kettle 6 in a molten condition through pipe 17 and is preferably placed in suitable containers to cool. However, removal of the finished grease from the kettle in a solid or semi-solid condition, as common in the prior art, is not precluded. Likewise the molten grease may be "fibred" by suitable stirring during the initial cooling stages if desired.

During the period of heating of the oil-fat mixture and during the subsequent saponification period, agitation by means of the paddles is preferably maintained continuously and a cooling agent such as water or brine is circulated through the cooling coil 30 at a predetermined rate and at a predetermined temperature, so as to effect a controlled dephlegmation of the vapors rising from the kettle. More or less oxidation may occur in the hot mass from the air overlying the liquid and from air which may be drawn into the kettle through the openings around the hinged cover, which is preferably not air-tight. By the operation of the steam jet 24 the vapors evolved from the kettle 6 are continuously drawn over to the condenser 21, and such condensate as may be developed in this condenser flows into the receiver 22. The condenser 21 may be cooled to any suitable condensing medium.

The following specific examples are given solely for the purpose of illustrating my invention and it is to be understood that I do not intend to limit the invention to the particular proportions, ingredients, or operating conditions recited.

In these specific examples the penetration or hardness of the resulting grease is expressed in each instance in terms of tenths of a millimeter penetration of an average sample as determined by the standard A. S. T. M. penetrometer and method for greases, and also with the same apparatus modified to the extent of having an extra weight of 475 grams placed within the penetrometer cone. Such latter reading will be termed "Penetration (modified A. S. T. M.)". This modified penetration test has been used on samples of relatively hard products to penetrate more deeply and give a higher and perhaps more accurate reading than that obtained with the same apparatus without the added weight.

A description of the A. S. T. M. penetration test may be found in reports of A. S. T. M. Committee D—2 on Petroleum products and lubricants and methods of test relating to petroleum products, under the heading "Tentative method of test for penetration of greases and petrolatum," test D—217—27—T.

Examples 1, 2 and 3

	1	2	3
Ingredients:			
Cylinder stock (secs. viscosity Saybolt at 210° F.)	176	176	176
Per cent	60.89	61.03	61.18
Hard tallow. Per cent	32.07	32.15	32.22
Flake caustic (98% NaOH). Per cent	3.52	3.41	3.30
Water. Per cent	3.52	3.41	3.30
Time of heating oil and fat mixture before saponification:			
Hrs. required to heat to 485° F.	3	4	2½
Hrs. maintained @ 485° F.	31	26½	21¾
Viscosity of mixture (secs. Saybolt at 210° F.):			
Before heating	99	100	100
After heating	165	154	140
Saponification with caustic soda solution (49% NaOH) consisting of flake caustic and water:			
Hrs. required to add solution	4½	4	4¾
Analysis of the final product—per cent. by weight:			
Tarry matter	3.75	2.50	1.50
Water	None	None	None
Sodium soap	30.80	30.90	31.30
Mineral oil	62.62	64.92	63.96
Free fatty acid (oleic)	1.51	0.70	2.41
Free fatty oil	0.86	None	0.55
Insoluble matter	0.44	0.80	None
Undetermined material	.02	.18	.28
Physical properties of final product:			
Melting point—°F.	348	366	337
Penetration (A. S. T. M. method)	41	47	84
Penetration (modified A. S. T. M.)	86	105	176

Examples 1, 2 and 3 demonstrate my invention by indicating the variation in hardness or penetration in the resulting greases by varying the time of maintaining the mixed oil and fat at high temperature. The same oil and fat are employed in approximately the same proportions in each example. In Example 1 the mixture of oil and fat was maintained at the high temperature for a period of 31 hours prior to saponification; in Example 2 the period of heating was 26½ hours, while in Example 3 it was 21¾ hours. The greases resulting from subsequent saponification, it will be observed, have modified A. S. T. M. penetrations of 86, 105 and 176, respectively.

Examples 4, 5 and 6

	4	5	6
Ingredients:			
Cylinder stock (secs. viscosity Saybolt at 210° F.)	176	176	176
Per cent	59.03	67.89	71.86
Hard tallow. Per cent	32.67	25.51	23.00
Flake caustic (98% NaOH). Per cent	4.15	3.30	2.57
Water. Per cent	4.15	3.30	2.57
Time of heating oil and fat mixture before saponification:			
Hrs. required to heat to 485° F	3	5	3½
Hrs. maintained @ 485° F	22	30	37½
Viscosity of mixture (secs. Saybolt at 210° F.):			
Before heating	95	109	110
After heating	133	187	210
Saponification with caustic soda solution (49% NaOH) consisting of flake caustic and water:			
Hrs. required to add solution	4½	4	4
Analysis of the final product—per cent. by weight:			
Tarry matter	0.52	2.84	3.40
Water	None	None	None
Sodium soap	34.00	24.00	20.40
Mineral oil	64.47	70.80	76.24
Free fatty acid	0.75	2.11	Neutral
Free fatty oil	None	None	None
Insoluble matter	None	None	None
Undetermined material	.26	.25	----------
Physical properties of final product:			
Melting point—°F	384	386	381
Pentration (A. S. T. M. method)	37	37	40
Penetration (modified A. S. T. M.)	39	86	89

The results of Examples 4, 5 and 6 show that by increasing the period of heating the mixed oil and fat the percentage of soap in the resulting greases may be reduced very considerably, while still maintaining practically the same hardness or penetration. These examples clearly illustrate the possibility of increasing the content of mineral oil lubricant and decreasing the soap content, thus lowering the cost of the final product while still maintaining the desired penetration or hardness.

Examples 7 and 8

	7	8
Ingredients:		
Cylinder stock (secs. viscosity Saybolt at 210° F.)	151	163
Per cent	37.37	39.78
Paraffin distillate (372 secs. viscosity Saybolt at 100° F.). Per cent	22.90	24.36
Hard tallow. Per cent	29.20	26.10
Stearic acid. Per cent	3.25	2.90
Flake caustic (98% NaOH). Per cent	3.64	3.43
Water. Per cent	3.64	3.43
Time of heating oil and fat mixture before saponification:		
Hrs. required to heat to 485° F	5	5
Hrs. maintained at 485° F	7	11
Viscosity of mixture (secs. Saybolt at 210° F.):		
Before heating	70	73
After heating	104	111
Saponification with caustic soda solution (49% NaOH) consisting of flake caustic and water:		
Hrs. required to add solution	4¾	5¼
Analysis of the final product—per cent. by weight:		
Free alkali	Acid	Acid
Water	None	None
Sodium soap	30.0	24.85
Mineral oil	59.95	66.22
Free fatty acid (oleic)	0.42	0.42
Free fatty oil	0.65	None
Undetermined material	8.98	8.51
Physical properties of final product:		
Melting point—°F	370	376
Penetration (A. S. T. M. method)	33	29
Penetration (modified A. S. T. M.)	81	75

Examples 7 and 8 resulted in relatively hard greases adapted primarily for the lubrication of locomotive driving journals. The mineral lubricating oil used consisted of cylinder stock and a paraffin distillate commonly known in the industry as "heavy red paraffin", the calculated viscosity of the mixed oils being about 90 and 93 seconds Saybolt at 210° F., respectively. The fats used were tallow and a small amount of stearic acid and, after heating the mixed fat and lubricating oil to a temperature of about 485° F. for the desired periods of time and saponification, greases were produced having nearly the same penetration, while the soap content by actual analysis was at least 5% lower in Example 8 as compared with Example 7.

These examples also indicate the preparation of greases adapted for the lubrication of driving journals of locomotives from lubricating oil containing a substantial amount of lower viscosity oil than cylinder stock, the latter having been used almost exclusively as the oil ingredient in the manufacture of prior art driving journal greases.

The foregoing greases made by practicing my invention are all relatively hard greases which may be used in place of other similarly hard greases made by the usual prior art methods and therefore containing larger quantities of soap and a lesser quantity of mineral oil. However, the invention is also well adapted to enable the manufacture of softer greases usually prepared from low viscosity mineral oils, such as soft cup greases, sponge greases, and the like, and which may contain lime, aluminum or other suitable soaps. The inclusion of such additional constituents as graphite, rosin, wax and the like, to impart certain specially desired physical properties to the product is not precluded.

Obviously, many modifications and variations of the invention as hereinbefore set forth may be made without departing from the spirit and scope thereof, and therefore only such limitations should be imposed as are indicated in the appended claims.

I claim:

1. The process of manufacturing grease comprising mixing mineral oil and fatty oil in proper proportion, subjecting the mixture to digestion by heating to a temperature of about 400–700° F. thereby producing a mixture of substantially greater viscosity than the original and forming reaction products in the mixture which impart hardness to the final grease, adding alkaline material to the digested mixture to react with its saponifiable constituents, and subjecting the evolved vapors to reflux condensation at such regulated and predetermined temperatures as to condense and return to the mass the valuable fatty acid content thereof.

2. The process of manufacturing grease comprising mixing mineral oil and fatty oil in the proper proportion, subjecting the mixture to digestion by heating to a temperature of about 400–700° F. thereby producing a mixture of substantially greater viscosity than the original and forming reaction products in the mixture which impart hardness to the final grease, adding alkaline material to the digested mixture to react with its saponifiable constituents, and subjecting the evolved vapors to reflux condensation at such regulated and predetermined temperature as to condense and return to the mass a uniform proportion of the valuable fatty acid content thereof as between successive batches.

GUS KAUFMAN.