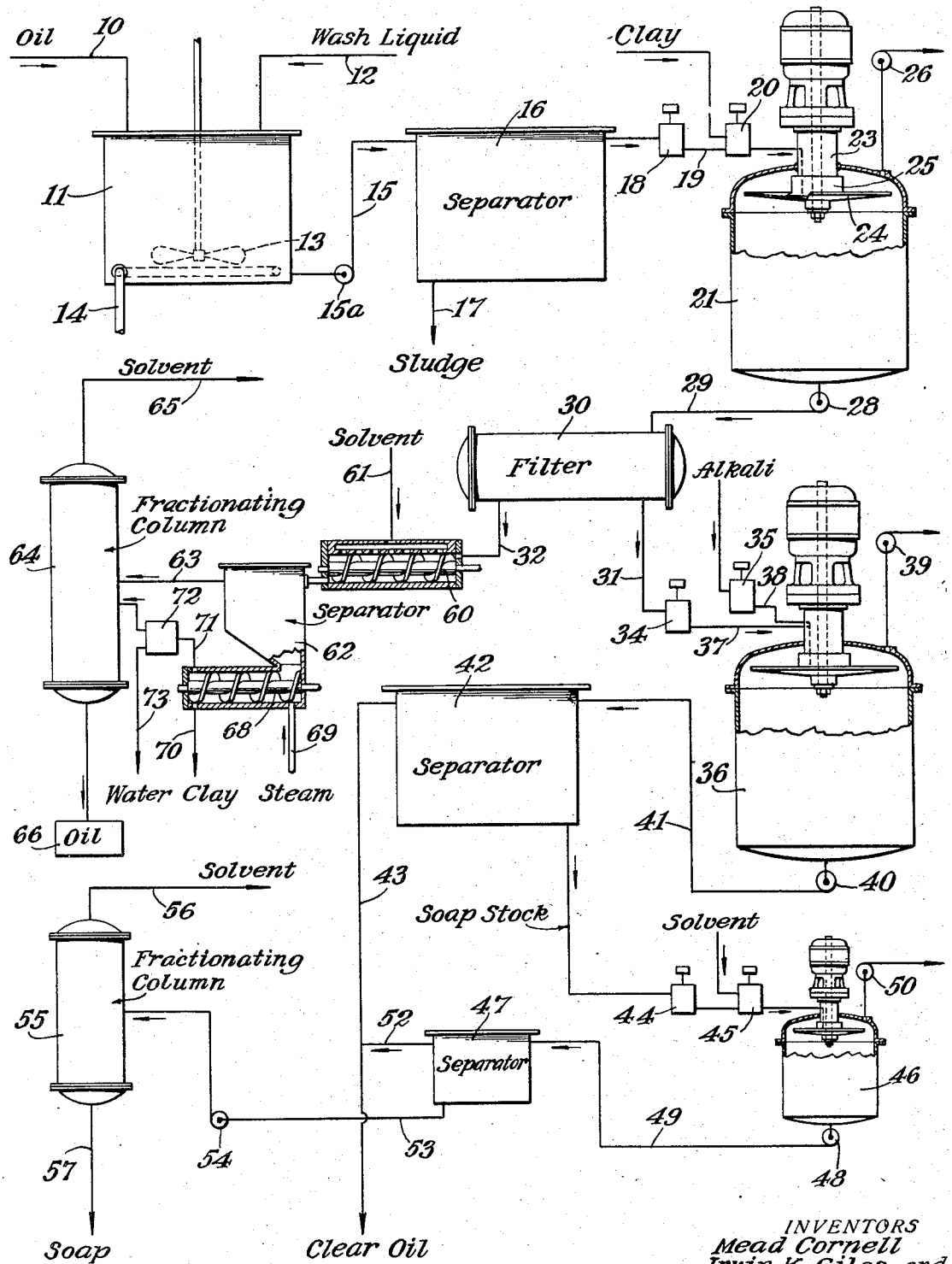
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REFINING OF COTTONSEED OIL

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REFINING OF COTTONSEED OIL

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16 Claims. (Cl. 260—425)

This invention relates to improvements in refining animal and vegetable oils, more particularly crude cottonseed oil.

Refining of crude oils of animal and vegetable origin by continuous methods has heretofore been proposed, but it has been found that the continuous treatment as practiced is costly as it can only be accomplished by the use of special apparatus and under the objectionable operating conditions of a minimum time cycle. This requires uneconomical quantities of reaction products and if the rapidity of reaction and separation is not maintained, inferior products result. The time factor is thus a critical operating condition.

One of the principal objects of the present invention is to provide an improved method for the refining of animal and vegetable oils, particularly cottonseed, soya bean and other glyceride oils, in which high production can be accomplished by a continuous flow of oil which is attenuated but not subdivided, in the presence of the treating materials in the respective treatment steps so that the amount of treating materials may be materially reduced.

A more specific object of the invention is to provide an improved manner of neutralizing the free fatty acids for removal thereof which comprises simultaneously mixing the oil body and neutralizing agent and immediately attenuating the mixture without subdivision or emulsification so that the soap will freely separate from the oil without critical time or temperature conditions.

Another object of the invention is to provide an improved method of refining vegetable and animal oils by which the end products are of a more highly refined quality, have a total value in excess of that of the end products heretofore produced, and in which the relatively low concentrations of oil carried by the treating materials can be recovered.

Further objects and advantages of the invention will appear from the following disclosure of a preferred form of embodiment of our invention taken in connection with the attached drawing which is a diagrammatic flow arrangement for the treatment of oils.

Oils of animal and vegetable origin, such as cottonseed oil, are commonly refined to be rendered edible, and deleterious matter, as well as free fatty acids, must be removed for this purpose. Such oils as prime crude oils may have impurities of approximately 1%, such impurities being of a gummy or mucilaginous type, and a free fatty acid content in the range of 1½% or more. In addition, there is usually a substantial amount of objectionable coloring matter. The removal of these elements, without excessive shrinkage, which now ranges from 5 to 9%, and without excessive use of time or materials, is essential for commercial acceptance. While the invention is not limited to any particular oil, and to no specific limit of impurities, the invention will be found especially applicable to the refining of such a product.

In accordance with the preferred form of embodiment of the invention, the gummy and mucilaginous matter is first removed by mixing the oil, which is carried by pipe 10 into tank 11, with a suitable washing liquid such as water entering at 12. Only a small proportion of wash water or liquid is required and with approximately 1% of impurities, the amount of water is approximately 5% of the incoming oil. The oil and wash liquid are slowly and gently stirred by the paddle 13 and, if desired, the entire mixture may be heated by suitable heating coils 14 to a temperature of approximately 110° F. The mixture of water and oil is then continuously pumped through pipe 15 by pump 15a (or carried by gravity if convenient) to a separating device such as settling tank 16 in which the oil separates from the gums and meal content which are mixed with the water. This sludge is preferably drawn off from the bottom, as at 17, and contains approximately all of the added water and all of the gummy resinous materials. This accounts for an unavoidable shrinkage of about 1% of the original feed.

As the second step in the process, the oil is preferably decolorized with clay or suitable adsorbent and substantial advantage is obtained in using a material such as Filtrol which is an acid treated, porous, silica of good decolorizing quality. The water washed oil is discharged in measured quantities, as for example, by a proportionating device 18, such as described in Cornell U. S. Patent No. 2,109,331, into a manifold 19. The clay is similarly fed in measured quantities into this manifold as by a proportionating device 20 of the type of proportionating device 18.

The clay, which may be in the order of 3% of the oil, is thus combined with oil and the mixture is then finely attenuated without subdividing the oil. This is preferably accomplished by the attenuating device 21, and one which is found especially satisfactory for this purpose is described in Cornell U. S. Patent No. 2,042,880. The flow of mixed oil and clay in such a device

is normally through the inlet housing 23 over the rotor 24, under the spreader 25, and it is then deflected downward by the shell so that a continuous thin film is maintained. The clay may be mixed at an elevated temperature if desired, and we find a temperature in the neighborhood of 140° F. to be desirable. The contacting is also preferably carried out under a substantial vacuum in the range of 26" of mercury obtained by vacuum pump 26, which assures an elimination of air and thus prevents oxidation. It is believed that operation under a suitable pressure of inert gas would also be effective.

The mixture of clay and oil which collects in the bottom of attenuating device 21 is then continuously removed by pump 28 and discharged through line 29 into the continuous filter 30. This filter is of any desired commercial type from which the filtrate is drawn off at 31 and the filter cake is removed at 32. The filtrate is a decolorified, gum free oil of a light pale color and is ready for the removal of the free fatty acids. It is found to be superior in color to the oils otherwise decolorized and the color adsorbing material is used in smaller quantities for the amount of coloring matter removed than under other conditions. This is due, to a substantial degree, to the finely attenuated stream which may be of a thinness of approximately .005", which assures a most intimate contact with the adsorbent.

The removal of the free fatty acids is accomplished by reaction with a suitable neutralizing agent and many are known, especially alkali such as caustic soda. In accordance with this process, a substantially complete removal of the free fatty acids can be accomplished without an excess of alkali, and therefore without saponifying the neutral oil. While we do not wish to be limited to any precise theory of operation, it appears to be possible to accomplish this step of the process by virtue of having first degummed and decolorized the oil, and then obtaining an effective film contact between the oil and alkali by attenuating the oil stream without subdividing the oil. This not only assures the desired neutralization, but avoids the formation of an emulsion, thereby facilitating the later separation of the oil from the soap.

The oil and alkali are mixed in precisely proportioned amounts as by proportionating devices 34 and 35 of the type heretofore described, except that the oil and alkali are simultaneously introduced into the attenuating device 36 through separate manifolds 37 and 38 to avoid premature contact before attenuation.

The attenuating device is similar in construction to the former one and is also preferably operated under a vacuum as produced by pump 39. Neutralization of the free fatty acids occurs during the period that the oil film is attenuated and complete neutralization occurs with the theoretically required amount of alkali. This is undoubtedly facilitated by the absence of all deleterious matter. If it is found desirable, a slight excess (5%) of alkali may be used. Accurate control of the time of contact and of the film thickness is had by variation of the rate of feed and the rotor speed.

The oil and soap in suspension are then drawn from attenuating device 36 by pump 40 and discharged through pipe 41 to a separating device 42 which may suitably be a settling tank. The free separation of the soap from the oil is a marked feature of the invention as it can be accomplished in a comparatively short time, merely by settling and without addition of heat. Approximately 95% of the incoming charge is discharged as a clear, gum free, acid free oil end product at 43, and this oil is so much improved in color and quality over the usual product, that it commands a premium in price.

The explanation for the free settling of the soap particles is not altogether established, although it is probably due to the fact that no emulsion exists but rather, the soap is suspended in the oil. The soap is relatively hard, being free of excess water, and it contains only a small amount (1% or less) of oil which can be recovered as hereinafter described. No saponification of the neutral oil occurs during the period of attenuation, and the alkali acts as a selective neutralizing agent only on the acid content of the oil. There appears to be no need of added heat, except to improve the viscosity of the product, and operations can be effectively carried out at normal room temperatures.

The soap stock which is removed from the bottom of separator 42, although substantially oil free, can be improved and the oil removed. A preferable arrangement is to introduce proportioned amounts of soap stock and a soap solvent such as alcohol and water through proportionating devices 44 and 45, and then attenuate the stream as by attenuating device 46, which is of a type similar to the prior devices 21 and 36, but substantially smaller in view of the much lower volume of materials handled. The intimate mixture of soap solution and oil is then separated as in any preferred form of separator such as the settling tank 47 into which the mixture is pumped by pump 48 in line 49. A vacuum is conveniently maintained on attenuating device 46 as by vacuum pump 50, and this prevents the formation of an emulsion. The clear oil which is drawn off at 52 represents approximately 1% of the original charge.

The soap and alcohol solution removed at 53 is pumped by pump 54 into fractionating column 55, which is operated under suitable conditions to remove the alcohol and water as soap solvent overhead at 56 and with the soap removed at 57 from the bottom. This soap is of especially high grade, being of superior color and free of oil, and it also commands a premium in the trade. Being free of oils, it is only approximately 3% or less of the initial charge of crude oil. This represents a definite shrinkage in the amount of refined oil, but is very much lower than ordinary.

Recovery of additional oil from the filter cake removed at 32 from filter 30 is also economically possible. This cake contains approximately 30% of oil (representing about 1% of the original charge), the clay and the adsorbed coloring matter removed in the filter. The clay is discharged through a form of spiral or other conveyor 60 in the presence of an oil solvent added at 61 to separator 62 which is conveniently a settling tank. The oil and solvent are removed from the separator through pipe 63 and discharged into fractionating column 64 in which the solvent which was used to free the oil from the clay is vaporized and removed overhead at 65. The oil is recovered at 66. If the solvent used also removes all of the entrained coloring matter from the clay, the oil will be of a very dark color and when sufficient has been accumulated, it may be run as a batch through the first attenuating device 21 by running it through proportionating device 18 and manifold 19. Approximately 1% additional yield may be obtained in this manner.

The clay from separator 62 is conveniently discharged into another conveyor 68 in the presence of steam or other vapor introduced at 69 to complete the removal of solvent. The clean clay is removed at 70 and the vapors conducted at 71 into the column 64. If necessary, a settling tank 72 may be used to separate out water as steam condensate, and this is drawn off at 73.

It is, of course, possible to add different solvents at 61 to remove the oil as a clear product, thus leaving the coloring matter in the clay for further treatment. In such case, the clear bleached oil recovered at 66 is approximately 1% of the oil recovered at 43 and 52. Clay revivification, if desired, would be carried out as a separate treatment in accordance with any desired plan.

During the entire process, the oil stream is never subdivided or broken up, but is maintained as a continuous body, and only attenuated during the treating steps so that the treating materials are active. This results in reactions which cannot be accomplished in any length of time of mixing in other machines and appears to explain the very rapid separation of the oil and soap after neutralization. Furthermore, the end products are clearly superior to the best possible under present commercial practices, and we not only use less clay and alkali, but reduce our shrinkage of which the principal part is an improved soap.

The separation of oil and other products in the various steps, although described as being effectively accomplished by settling, can also be accomplished by mechanical separation such as in a centrifuge, if desired. A preferred form of embodiment of the invention has been shown and it is well known that modifications may be made thereto without departing from the invention. A broad interpretation of the invention within the scope and spirit of the description herein and of the claims appended hereinafter is thus desired.

We claim:

1. An improved method for neutralizing the free fatty acids in animal and vegetable oils without any substantial saponification of the oil which comprises the steps of conducting separate streams of oil and a caustic neutralizing agent to a rotary disk mixing device, combining the separate materials on said disk, rotating said disk at a relatively high peripheral speed to cause said materials to flow across said disk in a thin film under substantial centrifugal force whereby immediate neutralization of the fatty acids is accomplished, removing the mixture of oil and neutralized acid in a continuous film into a liquid receiving zone without forming an emulsion of the oil, the amount of caustic neutralizing agent being slightly in excess of the amount theoretically required to neutralize the acid, the time of contact of neutralizing agent with the charge oil, the uniform dispersion of neutralizing agent with respect to the dispersion of the acid in the oil film and the amount of neutralizing agent being such that the acid is neutralized in preference to any reaction of the neutralizing agent with the oil.

2. An improved method for neutralizing the free fatty acids in animal and vegetable oils without any substantial saponification of the oil which comprises the steps of conducting separate streams of oil and a caustic neutralizing agent to a rotary disk mixing device, combining the separate materials on said disk, rotating said disk at a relatively high peripheral speed to cause said materials to flow across said disk in a thin film under substantial centrifugal force whereby immediate neutralization of the fatty acids is accomplished, removing the mixture of oil and neutralized acid in a continuous film into a liquid receiving zone without forming an emulsion of the oil, the amount of caustic neutralizing agent being slightly in excess of the amount theoretically required to neutralize the acid, the time of contact of neutralizing agent with the charge oil, the uniform dispersion of neutralizing agent with respect to the dispersion of the acid in the oil film and the amount of neutralizing agent being such that the acid is neutralized in preference to any reaction of the neutralizing agent with the oil, said neutralization being carried out at a substantially uniform temperature and under a subatmospheric pressure.

3. An improved method for refining animal and vegetable oils without substantial saponification of the oil which comprises the steps of mixing the oil with a small proportion of water to establish a hydration of the resinous matter therein, separating such hydrated resinous matter from the oil, conducting said oil to a rotary disk mixing device, separately conducting a stream of caustic neutralizing agent to said disk, combining the oil and neutralizing agent on said disk, rotating said disk at a relatively high peripheral speed to cause said materials to flow across said disk in a thin film under substantial centrifugal force whereby immediate neutralization of the fatty acids is accomplished, removing the mixture of oil and neutralized acid in a continuous film into a liquid receiving zone without forming an emulsion of the oil, the amount of caustic neutralizing agent being slightly in excess of the amount theoretically required to neutralize the acid, the time of contact of neutralizing agent with the charge oil, the uniform dispersion of neutralizing agent with respect to the dispersion of the acid in the oil film and the amount of neutralizing agent being such that the acid is neutralized in preference to any reaction of the neutralizing agent with the oil.

4. An improved method for refining animal and vegetable oils without substantial saponification of the oil which comprises the steps of mixing the oil with a small proportion of water to establish a hydration of the resinous matter therein, separating such hydrated resinous matter from the oil, conducting said oil with a proportionated amount of a decolorizing material to a rotary disk mixing device, rotating said disk to cause said materials to flow thereacross in a thin film to obtain intimate admixture of the decolorizing material with the color bodies in said oil, filtering out the decolorized oil, passing said decolorized oil to a second rotary disk, separately conducting caustic neutralizing agent to said second disk, rotating said second disk at a relatively high peripheral speed to cause said materials to flow across said disk in a thin film under substantial centrifugal force whereby immediate neutralization of the fatty acids is accomplished, removing the mixture of oil and neutralized acid in a continuous film into a liquid receiving zone without forming an emulsion of the oil, the amount of caustic neutralizing agent being slightly in excess of the amount theoretically required to neutralize the acid, the time of contact of neutralizing agent with the charge oil, the uniform dispersion of neutralizing agent with respect to the dispersion of the acid in the oil film and the amount of neutraliz-

ing agent being such that the acid is neutralized in preference to any reaction of the neutralizing agent with the oil.

5. An improved method for neutralizing animal and vegetable oils as claimed in claim 1 in which the neutralized fatty acids are solvent treated to remove additional oil, said oil being combined with the acid-free oil previously made.

6. An improved method for neutralizing animal and vegetable oils as claimed in claim 4 in which the neutralized fatty acids are solvent treated to remove additional oil, said oil being combined with the acid-free oil previously made.

7. The process of refining vegetable oils, which comprises treating said oils to remove gums contained therein, thereafter mixing a body of said oil with an alkali refining agent and settling the resulting soap stock from the oil to produce a soap stock-oil mixture substantially free of gums, withdrawing refined oil from said settled soap stock, and thereafter diluting the soap stock and separating additional oil from said soap stock.

8. A process of refining vegetable oils containing gums and free fatty acids, which comprises continuously mixing a degumming reagent with said oil to precipitate said gums and continuously centrifugally separating said gums from said oil, thereafter admixing a body of said oil with an alkali refining reagent and settling the resulting soap stock from said oil, withdrawing refined oil from said settled soap stock and admixing the remaining soap stock containing entrained oil with a diluting agent, and separating oil from said soap stock.

9. The process of refining vegetable oils, which comprises treating said oils to remove gums contained therein, thereafter mixing a body of said oil with an alkali refining reagent and settling the resulting soap stock from the oil to produce a soap stock-oil mixture substantially free of gums, withdrawing refined oil from said settled soap stock, and thereafter diluting the soap stock with water and separating additional oil from said soap stock.

10. The process of refining vegetable oils, which comprises treating said oils to remove gums contained therein, thereafter mixing a body of said oil with an alkali refining agent and settling the resulting soap stock from the oil to produce a soap stock-oil mixture substantially free of gums, withdrawing refined oil from said settled soap stock, and thereafter diluting the soap stock with water to form a flowable mixture and continuously centrifugally separating additional oil from said soap stock.

11. The process of refining vegetable oils, which comprises subjecting an oil which is substantially free of gummy material to treatment including admixing a body of said oil with an alkali refining reagent and settling from said oil a soap stock substantially free of gummy material, and thereafter diluting the soap stock to form a flowable mixture and separating additional refined oil from said soap stock.

12. The process of refining vegetable oils, which comprises subjecting an oil which is substantially free of gummy material to treatment including admixing a body of said oil with an alkali refining reagent and settling from said oil a soap stock substantially free of gummy material, and thereafter diluting the soap stock with water to form a flowable mixture and continuously centrifugally separating additional oil from said soap stock.

13. A process of refining vegetable oils containing gums and free fatty acids, which comprises continuously mixing a degumming reagent with said oil to precipitate said gums and continuously centrifugally separating said gums from said oil, thereafter admixing a body of said oil with an alkali refining reagent and settling the resulting soap stock from said oil, withdrawing refined oil from said settled soap stock and admixing the remaining soap stock containing entrained oil with a diluting agent and continuously centrifugally separating additional refined oil from said soap stock.

14. A process of refining vegetable oils containing gums and free fatty acids, which comprises continuously mixing a degumming reagent with said oil to precipitate said gums and continuously centrifugally separating said gums from said oil, thereafter admixing a body of said oil with an alkali refining reagent and settling the resulting soap stock from said oil, withdrawing refined oil from said settled soap stock and admixing the remaining soap stock containing entrained oil with sufficient water to form a flowable mixture and continuously centrifugally separating additional refined oil from said soap stock.

15. The continuous process of treating vegetable oil containing phosphatides, non-fatty matter and free fatty acids, comprising the steps of mixing a stream of water which will precipitate said phosphatides and non-fatty matter with a stream of oil in sufficient amount and concentration to substantially completely precipitate the same without destruction of said phosphatides or said oil, promptly continuously centrifugally separating the precipitated material from the oil, promptly mixing a stream of an alkaline refining reagent with a stream of the resultant oil in sufficient amount and concentration to substantially completely react with the fatty acids therein to form soap stock, and promptly thereafter centrifugally separating said soap stock from said oil to produce a substantially pure soap stock.

16. A quick and continuous process for treating vegetable oils containing gums, which comprises the steps of: continuously admixing water as a precipitating reagent in an amount sufficient to precipitate said gums but without substantially reacting with the fatty acids contained therein; promptly, before injury to the oil, continuously centrifugally separating the precipitated gums from said oil; promptly continuously mixing a neutralizing reagent with the thus degummed oils in sufficient amount to substantially completely neutralize the free fatty acids remaining therein and to form foots and promptly continuously centrifugally separating the foots from the thus refined oil.

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CERTIFICATE OF CORRECTION.

Patent No. 2,337,041. December 21, 1943.

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It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, first column, lines 20 and 21, for "decolorified" read --decolorized--; page 4, second column, line 57, claim 16, after "the" insert --free--; 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 29th day of February, A. D. 1944.

Leslie Frazer

(Seal) Acting Commissioner of Patents.