UNITED STATES PATENT OFFICE.

FRIEDRICH BERG, OF CLEVELAND, OHIO.

PROCESS OF REFINING PETROLEUM.

SPECIFICATION forming part of Letters Patent No. 623,066, dated April 11, 1899.

Application filed November 5, 1896. Renewed September 17, 1898. Serial No. 691,167. (No specimens.)

To all whom it may concern:

Be it known that I, FRIEDRICH BERG, of Cleveland, Cuyahoga county, Ohio, have invented certain new and useful Improvements in the Process of Refining Petroleum; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it pertains to make and use the same.

My invention relates to improvements in refining petroleum or hydrocarbon oils that contain sulfurous compounds or impurities.

For several years I have carried on experiments with a view of removing sulfurous compounds and other impurities from petroleum, and on the 19th day of May, 1896, United States Letters Patent No. 560,463, involving a process of refining petroleum, were granted upon an application filed by me April 31, 1893. Since the filing of said application I have been very active in experimenting in the line of refining petroleum with a view of obtaining a superior grade of illuminating-oil at a much less cost, and I have attained the desired end in devising the process hereinafter described and claimed.

In carrying out my improved process I place the crude oil—that is, oil in the state in which it is taken from the earth—(and I have employed the most inferior grade of oil) in a tank or receptacle and add alkali, preferably caustic soda, thereto and stir and mix the alkali and crude oil together in any approved manner. The alkali is of the strength ordinarily employed in the treatment of petroleum-oils, and said alkali and crude oil are preferably mixed at the ordinary temperature. A mixture in the proportions of about one gallon of alkali to about one barrel of the oil is preferable. I then heat the mixture of alkali and crude oil to about 200° Fahrenheit or somewhat greater temperature and keep the mixture heated at this temperature for a few hours. A heat of 200° Fahrenheit is preferable and the maintenance of the mixture at this temperature for about five hours is desirable. The test by which it may be determined when the heating of the mixture at a temperature of 200° Fahrenheit has proceeded sufficiently is the great discoloration of the oil. The alkali acts upon the impurities or foreign compounds and destroys the said compounds, thereby resulting in a heavy discoloration of the oil. The length of time that the oil is heated to the aforesaid temperature will depend upon the nature of the impurities, and it will be perceived that it is not absolutely necessary to heat the oil to 200° Fahrenheit, in which case, however, the heating would likely have to continue for a longer period of time. Of course it is obvious that the lighter products of petroleum—such, for instance, as naphtha—will vaporize at this temperature; but these escaping vapors can be saved by any well-known apparatus suitable for the purpose, and the escaping products can be returned, if desired, to the body of the oil that requires further treatment. The mixture of crude oil and alkali at the aforesaid temperature should be stirred for about ten minutes every hour during the time in which the mixture is maintained at the comparatively high temperature indicated. The agitation of the mixture at intervals, as aforesaid, facilitates the work of the alkali that is instrumental in decomposing the sulfurous compounds or foreign matter contained in the oil and is also instrumental in effecting the destruction or removal of water contained in the oil. I would here remark that all crude oil contains water in an undesirable quantity. The quantity of water contained in the oil is generally about five per cent. All water should be removed from the oil, if possible, in order to maintain a good illuminating-oil. During this treatment of the crude oil with the alkali most of the impurities and water contained in the oil are precipitated or destroyed. Of course this treatment does not entirely free the oil from impurities and water, and further treatment of the oil is therefore required.

After the treatment of the oil with the alkali in the manner hereinbefore described I remove the oil from the tank or still, and consequently from the alkali and foreign matter that have been precipitated or have settled in said tank or still. The remaining alkali can be used in the treatment of another quantity of raw crude oil. I then place the oil that has been removed from the alkali in another tank, wherein it is kept for a few days, preferably six days, at a moderate temperature, (about 70° Fahrenheit,) and during this

time the alkali still remaining in the oil and that did not settle in the tank or still wherein the oil was treated with the alkali at the aforesaid comparatively high temperature continues its work of destroying sulfurous compounds or impurities and water still contained in the oil.

No expensive chemicals are required in my improved process to accomplish the destruction or precipitation of the objectionable foreign matter and water, and I have found that the work of the alkali still contained in the oil for several days is invaluable in effecting the purification and deodorization of the oil.

The oil being treated is next removed from the tank wherein it was lastly undergoing treatment to a still and is there subject to the ordinary process of distillation. The distillation of the oil after the removal of the larger percentage of the sulfurous compounds is important, because it is obvious that a distilling apparatus will last much longer if the larger percentage of the sulfurous compounds that would be highly destructive to said apparatus is removed before the distillation. The distillate obtained could be used as and is an illuminating-oil, but has not all the properties of what I consider first-class illuminating-oil and requires further refinement in order to make it a first-class illuminating-oil. A small amount of alkali is still in the oil during the distillation just referred to, and this remaining alkali does effective work in destroying the lighter sulfurous compounds or impurities still contained in the oil. The distillate obtained is then treated lightly with sulfuric acid of the usual commercial strength. This acid removes the water contained in the distillate. If fresh acid is used, I prefer about three pounds of the acid to one barrel of the distillate. If spent acid is employed, I prefer about five pounds of the acid to about one barrel of the distillate. Spent acid would of course be generally employed. The acid and oil are mixed at the ordinary temperature and are agitated or stirred in any approved manner for about a half-hour. The oil should not be washed after the treatment of the distillate with the acid, as has heretofore been practiced, because I have found that the washing of the oil with water is attended by the liberation of hydrogen from the water and acid, and this hydrogen and the sulfur in the acid and the impurities still contained in the oil form new sulfurous compounds, and the oil instead of being cleaned is really made more impure. I would call particular attention to the importance of removing as far as possible every trace of water contained in the oil. After having treated the distillate with the acid, as hereinbefore described, I again treat the oil with an alkali in any approved manner for the purpose of neutralizing and destroying the acid and impurities. I employ preferably about one gallon of the alkali to one barrel of the oil and mix or stir the same together in a tank for about a half-hour. After this second treatment of the oil with an alkali I remove the oil into a still and again distil. Any vapors escaping in this second distillation can, it is obvious, be saved by well-known apparatus suitable for the purpose. After the second distillation the oil, now a second distillate, is subject to a light treatment with sulfuric acid, and in this treatment of the oil with acid about two pounds of fresh acid are preferably used to about one barrel of the oil. The function of this last treatment of the oil with acid is to remove from the oil or second distillate any water that is still contained in the oil. The resulting product is an oil that is exceedingly clean, that is odorless, that has superior illuminating properties, and that costs much less, it will be observed, than the illuminating-oils obtained by the processes of refinement heretofore practiced or invented.

What I claim is—

1. The petroleum-refining process herein described, comprising, first, a treatment of the crude oil with an alkali at a comparatively high temperature, maintaining this mixture at said temperature for several hours and stirring or agitating the mixture at intervals during this time; then, removing or separating the alkali and foreign matter that has been precipitated or settled during said alkali treatment and permitting the alkali still contained in the oil to stand for several days at a moderate temperature and continue its work of destroying sulfurous compounds and impurities still remaining in the oil; then, distilling the oil, and finally, treating the distillate with sulfuric acid for the purpose of removing the water contained in the distillate, substantially as hereinbefore described.

2. The petroleum-refining process herein described, comprising, first, a suitable treatment of the crude oil with an alkali at a comparatively high temperature and maintaining the mixture at such temperature for several hours; secondly, the removal or separation of the oil from the alkali and foreign matter that have been precipitated or have settled during said alkali treatment, and permitting the alkali still remaining in the oil to continue its work for several days at any suitable temperature; thirdly, distilling the oil; fourthly, subjecting the distillate to a suitable treatment with sulfuric acid; fifthly, again suitably treating the oil with an alkali for the purpose of neutralizing and removing the acid and impurities; sixthly, again distilling the oil, and seventhly, subjecting the second distillate to a suitable treatment with sulfuric acid, in order to remove all water still contained in the oil, substantially as hereinbefore described.

3. The petroleum-refining process herein described, comprising, first, the mixing of an alkali with the crude oil in suitable proportions, heating this mixture to a comparatively high temperature, maintaining this

mixture at a comparatively high temperature for several hours and stirring or agitating the mixture at suitable intervals during this time; secondly, removing or separating the oil from the alkali and foreign matter that have been precipitated or have settled in the aforesaid alkali treatment and permitting the oil, that still contains alkali, to stand for several days at a moderate temperature; thirdly, distilling the oil; fourthly, subjecting the distillate to a light treatment with sulfuric acid; fifthly, again treating the oil with an alkali for the purpose of neutralizing and removing the acid and impurities; sixthly, again distilling the oil, and, seventhly, subjecting the second distillate to a light treatment with sulfuric acid to remove all water still contained in the oil, substantially as set forth.

4. The petroleum-refining process herein described, comprising, first, the mixing of an alkali with crude oil in suitable proportions, heating this mixture to about 200° Fahrenheit and keeping the mixture heated at this temperature for a few hours, and stirring the mixture for a short time every hour during this treatment; secondly, separating the oil from the alkali and foreign matter that have been precipitated or have settled during the treatment of the oil with the alkali, and then permitting the alkali still contained in the oil to do work for several days at a moderate temperature; thirdly, distilling the oil; fourthly, subjecting the distillate to a light treatment with sulfuric acid; fifthly, again treating the oil with an alkali; sixthly, again distilling the oil, and, seventhly, subjecting this second distillate to a light treatment with sulfuric acid, substantially as set forth.

5. The petroleum-refining process herein described, comprising, first, the mixing of an alkali with crude oil in suitable proportions, heating this mixture to a comparatively high temperature and keeping the mixture heated at this temperature for about five hours, and stirring the mixture for a short time every hour during said alkali treatment; secondly, separating the oil from the alkali and foreign matter that have been precipitated or have settled during the aforesaid treatment of the oil with the alkali, and then permitting the alkali still contained in the oil to continue its work for several days at a moderate temperature; thirdly, distilling the oil; fourthly, subjecting the distillate to a light treatment with sulfuric acid in order to neutralize and destroy the acid and impurities; fifthly, again treating the oil with an alkali; sixthly, again distilling the oil, and seventhly, subjecting the second distillate to a light treatment with sulfuric acid for removing all water still contained in the oil, substantially as set forth.

6. The petroleum-refining process herein described, comprising, first, the mixture of an alkali with crude oil in suitable proportions, heating this mixture to a comparatively high temperature and keeping the mixture heated at this temperature for a few hours, and stirring the mixture for a short time every hour during this treatment; secondly, separating the oil from the alkali and foreign matter that have been precipitated or have settled during the treatment of the aforesaid oil with the alkali, and then permitting the alkali still contained in the oil to continue its work for about six days at a moderate temperature; thirdly, distilling the oil; fourthly, subjecting the distillate to a light treatment with sulfuric acid in order to neutralize and destroy the acid and impurities; fifthly, again treating the oil with an alkali; sixthly, again distilling the oil, and seventhly, subjecting the second distillate to a light treatment with sulfuric acid to remove all water still contained in the oil, substantially as set forth.

7. The petroleum-refining process herein described, involving, first, the mixture of the alkali with crude oil in the proportions of about one gallon of the alkali to one barrel of the oil and heating this mixture to about 200° Fahrenheit, and maintaining the mixture at this temperature for a few hours, and stirring and agitating the mixture during this time for about ten minutes every hour; secondly, separating the oil from the impurities and alkali that have been precipitated or have settled during the preceding alkali treatment, and permitting the alkali that is still contained in the oil to continue its work for several days at a moderate temperature; thirdly, distilling the oil; fourthly, subjecting the distillate to a light treatment with sulfuric acid in order to neutralize and destroy the acid and impurities; fifthly, again treating the oil with an alkali; sixthly, again distilling the oil, and, seventhly, treating the second distillate lightly with sulfuric acid to remove all the water still contained in the oil, substantially as set forth.

In testimony whereof I sign this specification, in the presence of two witnesses, this 31st day of October, 1896.

FRIEDRICH BERG.

Witnesses:
C. H. DORER,
ELLA E. TILDEN.