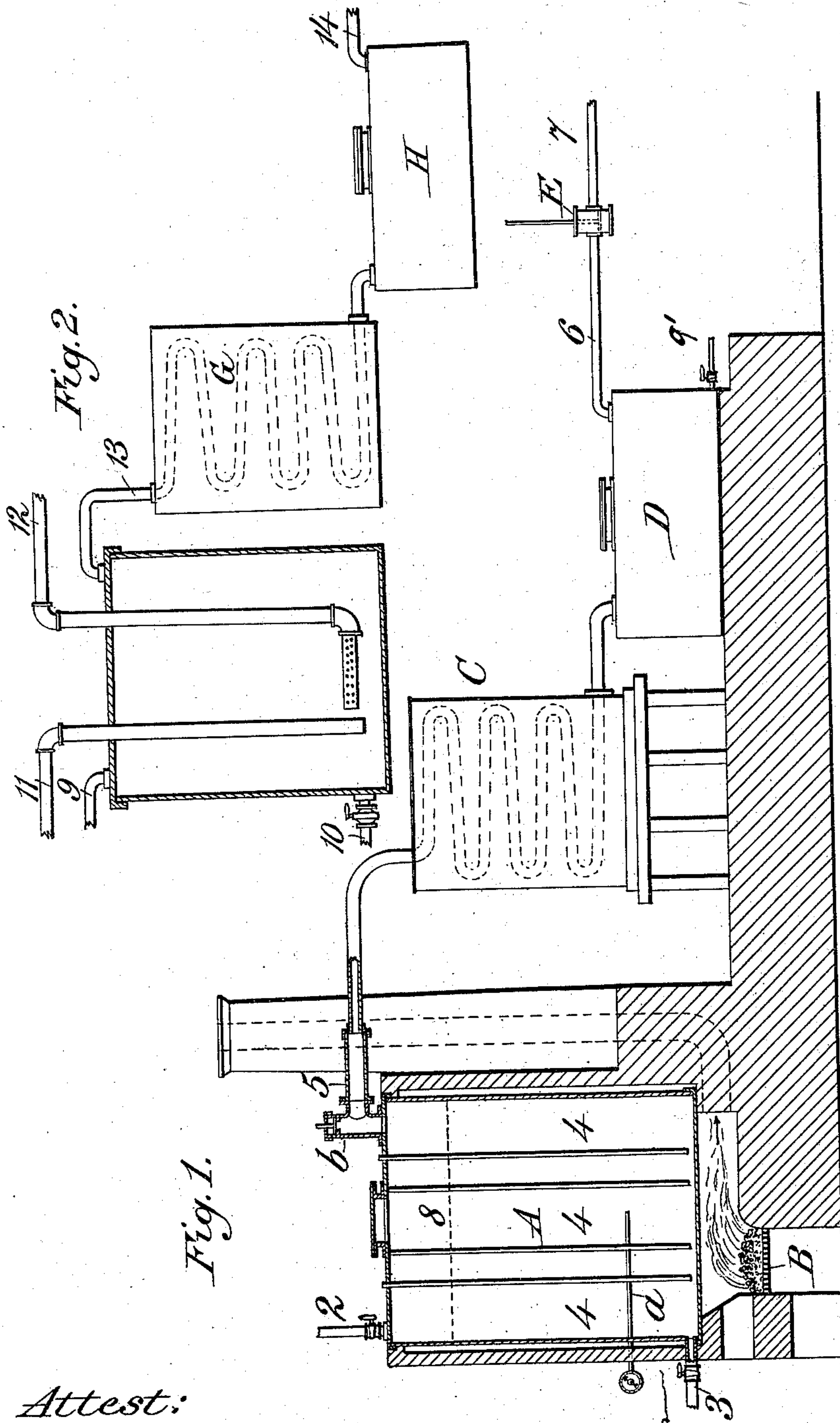


(No Model.)

F. X. BYERLEY.
PROCESS OF REFINING LIMA OIL.

No. 547,329.

Patented Oct. 1, 1895.



Attest:

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UNITED STATES PATENT OFFICE.

FRANCIS X. BYERLEY, OF CLEVELAND, OHIO.

PROCESS OF REFINING LIMA OIL.

SPECIFICATION forming part of Letters Patent No. 547,329, dated October 1, 1895.

Application filed July 18, 1894. Serial No. 517,889. (No specimens.)

To all whom it may concern:

Be it known that I, FRANCIS X. BYERLEY, a citizen of the United States, residing at Cleveland, in the county of Cuyahoga and State of Ohio, have invented certain new and useful Improvements in Refining Ohio or Lima Petroleum; and I do hereby declare the following to be a full, clear, and exact description of the same.

This invention relates to the removal from petroleum containing the same of those impurities which are characteristic of the oil found in the vicinity of Lima, Ohio, and which, as is well known, make impracticable the refining of such oil by the so-called "Pennsylvania" or "sulphuric-acid and alkali" treatment, long used successfully upon oil from the Pennsylvania oil-fields. These impurities are generally understood to be of a sulphurous nature and are known to be radically different in kind from the impurities of Pennsylvania oil. Moreover, while their precise chemical constitution is not known, they are easily recognizable by those skilled in the art. The oil which contains them is very generally known as "Ohio" oil or as "Lima" oil, not because it is the only oil produced in Ohio, nor because it is found only at Lima, or even only in Ohio, but as a convenient designation to distinguish the same from Pennsylvania oil or oil such as is derived from the Pennsylvania fields. So far as I am aware, all the existing wells in the vicinity of Lima, Ohio, furnish oil containing these characteristic impurities. It is not considered necessary here to detail the peculiar known properties by which they are recognizable; but it may be observed that on subjecting the same to the action of air, as hereinafter described, they impart a yellow coloration to the oil containing them, whereas a similar treatment of impure Pennsylvania oil develops a reddish coloration. The designation, therefore, of "Ohio" or "Lima" oil in this specification will be understood to indicate the kind of oil, or rather the kind of impurities in the oil, whether the oil be found at Lima or elsewhere in Ohio, or in places outside of Ohio, as in Canada, for example, where the oil is similar.

In accordance with the present invention, the oil containing the characteristic impurities of Ohio or Lima oil is (by the aid of an

exhaustion of the vapors from above the oil) distilled at a low temperature, below the temperature of ordinary distillation, and during such distillation the said impurities are subjected to the action of air or analogous agent in volume or quantity sufficient to effect a substantial conversion of said impurities into substances which are either more volatile than the hydrocarbons comprising the burning oil, and therefore removable by steam stilling or analogous operation, or are soluble in sulphuric acid, water, or alkaline lye, and are therefore removable by the well-known treatment with sulphuric acid, water, and alkali. The action of the air is believed to be one of oxidation. So far as I am aware, while the general idea of applying oxidation to these impurities is not new, it was never before known that they could be acted upon as hereinbefore indicated, nor was it known how to effect such action. I attach much importance to the distillation at a low temperature, it being in fact essential, since an undue heat (such as would be required if air were forced into an ordinary still) interferes with the desired conversion, either by neutralizing the influence of the air or by perverting its influence, so that compounds result which are not readily removable, but become fixed in the oil.

It is advantageous and a special improvement to agitate the oil in the still during the distillation at a low temperature, which agitation is best effected by the inflow of streams of air into the body of oil, induced by the exhaustion of the vapors in the still from above the oil. Such air acts both mechanically to agitate the oil and chemically in attacking the peculiar impurities therein.

The distillate from the preceding operation is deprived of light products by heat and an aeriform fluid, (steam stilling or analogous operation,) and the thus treated residue is then finished by the sulphuric acid and alkali or equivalent treatment. While a useful result may be accomplished in this stage without aeration, as by injecting live steam without air into the distillate from the former treatment, it is an advantage and a special improvement to aerate such distillate by injection of air with the live steam or without steam, the distillate in the latter case being heated, but carefully, so as not to succeed, say, 250° Fahrenheit.

The injection of air and live steam is considered the best mode, the steam heating the oil to the desired temperature and the air attacking any partially-converted impurities.

- 5 The still does not require to be heated otherwise than by the injected steam.

The accompanying drawings, which form part of this specification, illustrate an apparatus suitable for use in carrying out the new
10 or improved process.

Figure I is a view of the still with its condenser and receiver, and Fig. II a view of the distillate aerating and steaming vessel with its condenser and receiver.

- 15 The still A, of ordinary construction, has the pipe 2 for the introduction of the oil to be distilled, the pipe 3 for drawing off the residuum, a number of pipes 4 for introducing air, and the gooseneck 5 for the escape of the oil-
20 vapors, and it is also provided with a fireplace B, a pyrometer *a*, and a safety-valve *b* (not loaded). The pipes 2 and 3 have each a stop cock or valve. The air-inlet pipes 4 extend inside the still nearly to the bottom and are
25 open at both ends. The gooseneck 5 is connected with the condensing-worm C, and the outlet of this worm is connected with the receiver D, whose upper part is connected by air-pipe 6 with the suction of a double-acting
30 pump E. The pipe 7 is an escape for the air and uncondensed vapors from the receiver D. If crude oil is to be distilled, there may be separate receivers for the different products, as naphtha, burning oil, &c., although it is
35 preferred to collect all the products in one receiver until the burning oil has been separated and only a heavy residuum is left in the still.

- The still A is filled, say, to the dotted line 8,
40 with the crude Ohio or Lima oil, although the height to which the still is filled is not important, and the fire is started in the chamber B, so as to raise the oil in the still to a distilling temperature. At the same time the
45 pump E is operated, so as to draw air from outside into the still. This air rises through the heated oil and mixes with the vapors produced by the heat, and the mixture of air and oil-vapors is drawn into and through the con-
50 denser C, where the petroleum vapors are condensed to a liquid and the air is liberated therefrom. Both the air and the liquid oil enter the receiver D.

- The following figures may be used as a
55 basis of working, it being understood that the invention is not restricted to them, and that they are to be followed and applied, not slavishly, but with the usual skill of the art.

- For a still ten feet high and eight feet in
60 diameter there may be five one-inch air-inlet pipes 4 and a double-acting air-pump E of eight inches diameter of piston and twelve-inch stroke. The still being filled about eight-
65 tenths full of crude Ohio oil, the pump E may be run at the rate of sixty-six revolutions (or complete reciprocations) more or less per minute, and fire in chamber B regulated to give

good run of distillate—say one hundred gallons per hour. During the distillation the distillate is examined, test portions being
70 drawn off, say, at the pipe 9 from time to time. If a test portion is not free from the characteristic odor of Ohio oil, the action of the pump is accelerated, so as to secure a larger
75 flow of air into the still. If a test portion of the distillate indicates scorching or burning, (and it may be observed that the oil assumes a burned condition at a lower temperature
80 than in ordinary distillation,) the fire in the chamber B should be slackened. If the temperature falls too low, the stoppage of distillation will indicate that the fire should be in-
85 creased. By properly regulating the inflow of air and the heat the burning oil and lighter products can be run off in about twenty hours and the residuum be then drawn off by the
90 pipe 3. During the distillation the temperature in the still will be gradually raised as heavier products are vaporized. The lighter products come over at a temperature of from 150° to 160° Fahrenheit, and the temperature
95 is gradually raised. At 400° to 450° Fahrenheit the last of the burning oil will generally distill over, while with ordinary distillation a temperature of about 700° Fahrenheit is neces-
sary. The agitation of the oil during the run is advantageous in assisting the liberation
100 of the oil-vapors. The air introduced by the pipes 4 operates mechanically as an agitator of the oil and chemically as a converter of the impurities into more readily removable sub-
stances.

It is not necessary, although considered preferable, to rely wholly on an exhaust to
105 draw the air into the still, as the air may be compressed by a pump or otherwise before it enters the still. However, if it be attempted simply to force the air into the still, the effect
110 is, as I have found, to prevent the alteration, which I have above indicated, of the peculiar compounds of Ohio oil by compelling the use
115 in the distillation of a high temperature. If, therefore, artificial pressure is applied to the air before entry, care should be taken to supply a sufficient exhaust to enable the distilla-
120 tion to go on at a sufficiently low temperature to effect the necessary conversion or alteration of the impurities. After the burning oil has collected in the receiver D the entire con-
125 tents of the latter (including gasoline, naphtha, and burning oil) are then transferred to a closed vessel provided with appliances for steaming and aerating the distillate, with a pipe in its bottom furnished with a stop-cock
130 to draw off the contents of the vessel, and also connected with a condenser to condense and recover the vaporized portion. In Fig. II the vessel for receiving the distillate is shown
provided with a filling-pipe 9, a draw-off 10, a steam-inlet pipe 11, and an air-inlet pipe 12,
135 and having its vapor-space connected by the pipe 13 with a condenser G. The vessel H receives the condensed products and has an escape-pipe 14 for the uncondensed products.

The receiver D could be provided with these appliances, if desired. In this vessel the water produced by the before-described distillation is allowed to settle, and is then carefully drawn off through the pipe in the bottom. Live steam of, say, sixty pounds pressure per square inch is then injected into the oil with, say, about an equal amount of air through a nose or perforated pipe inserted therein. This steaming and aerating is continued until the residual or burning oil in the vessel is not affected by a solution of plumbate of soda. This can be ascertained by withdrawing a test portion from the vessel and treating it with the solution. When no discoloration of the oil ensues from the treatment, the operation is complete. Should, however, the fire test and gravity of the oil at this stage not be what are desired, the operation may be continued till these points are attained. Those skilled in the art will understand how to regulate the flow of steam to drive off the lighter products and leave the burning oil. During such operation the air will act upon the impurities which may have come over in a partly-altered condition, and thus effect (in co-operation with the prior treatment) a more thorough purification. A tolerably satisfactory result can be obtained without this aeration of the distillates, steam alone being used to drive off the light products, and, as already mentioned, aeration could be effected and light products driven off by injecting air at a suitable temperature without the injection of steam.

The burning oil, after the two treatments described, is agitated with sulphuric acid and washed with water and soda-lye. This finishes the oil according to the present invention. The oil thus finished will be found freed substantially from the peculiar impurities characteristic of Ohio or Lima oil, and suitable for burning in lamps. Should the further treatment of any known or suitable description be thought advisable, it may of course be applied; but its use or disuse is something outside of the present invention.

In a concurrent application, on which Letters Patent, Serial No. 524,130, were granted to me August 7, 1894, I have described and claimed (among other things) a process in which petroleum or pitch-forming oil (including tar) is heated in a still with exhaustion of the products of distillation, agitation of the oil, and exposure to air, the temperature of said oil being gradually increased during the distillation to a pitch-forming non-cooking temperature and continued at such temperature until a solid or product solidifying on cooling is obtained.

My present process does not result in a solid or product solidifying on cooling, but in certain purified distillates, and is not even attended by the formation of a residual product which is solid or solidifies on cooling, although, of course, the fluid residuum of my present process is convertible by a subsequent procedure

into such a product. The use which is made of said fluid residuum, whether by forming a solid or solidifying product therefrom or by employing it in other ways, is a matter that does not concern the present invention.

I claim as my invention or discovery—

1. The process of obtaining purified distillates from Ohio or Lima oil, or in other words from oil containing the impurities which are characteristic of the petroleum from the vicinity of Lima, Ohio, and which resist removal by the sulphuric acid and alkali or so-called Pennsylvania treatment, the said process consisting in distilling such oil at a low temperature with the aid of an exhaustion of the vapors, and during such distillation subjecting the said impurities to the action of air in volume sufficient to effect a substantial conversion thereof, as indicated by the loss of the characteristic malodor of the aforesaid oil, as set forth.

2. The process of obtaining purified distillates from Ohio or Lima oil, or in other words from oil containing the impurities which are characteristic of the petroleum from the vicinity of Lima, Ohio, and which resist removal by the sulphuric acid and alkali or so-called Pennsylvania treatment, the said process consisting in distilling such oil at a low temperature with the aid of an exhaustion of the vapors, and during such distillation agitating the oil and subjecting the said impurities to the action of air in volume sufficient to effect a substantial conversion thereof, as indicated by the loss of the characteristic malodor of the aforesaid oil, as set forth.

3. The process of obtaining purified distillates from Ohio or Lima oil, or in other words from oil containing the impurities which are characteristic of the petroleum from the vicinity of Lima, Ohio, and which resist removal by the sulphuric acid and alkali or so-called Pennsylvania treatment, the said process consisting in distilling such oil at a low temperature with the aid of an exhaustion of the vapors, and during such distillation introducing streams of air into the body of oil in distillation, thus by the same air agitating the oil and subjecting the said impurities to the action of air in volume sufficient to effect a substantial conversion thereof, as indicated by the loss of the characteristic malodor of the aforesaid oil, as set forth.

4. The process of obtaining purified distillates from Ohio or Lima oil, or in other words from oil containing the impurities which are characteristic of the petroleum from the vicinity of Lima, Ohio, and which resist removal by the sulphuric acid and alkali or so-called Pennsylvania treatment, the said process consisting in distilling such oil at a low temperature with the aid of an exhaustion of the vapors, during such distillation subjecting the said impurities to the action of air in volume sufficient to effect a substantial conversion thereof, as indicated by the loss of the characteristic malodor of the aforesaid oil, and af-

terward driving off the light products with the aid of an aeriform fluid, and subjecting the resulting or burning oil residue to the sulphuric acid and alkali treatment, as set forth.

5 5. The process of obtaining purified distillates from Ohio or Lima oil, or in other words from oil containing the impurities which are characteristic of the petroleum from the vicinity of Lima, Ohio, and which resist removal

10 by the sulphuric acid and alkali or so-called Pennsylvania treatment, the said process consisting in distilling such oil at a low temperature with the aid of an exhaustion of the vapors, during such distillation subjecting the

15 said impurities to the action of air in volume sufficient to effect a substantial conversion thereof, as indicated by the loss of the characteristic malodor of the aforesaid oil, and afterward driving off the light products and

20 aerating the resulting or burning oil residue, and subjecting such aerated residue to the sulphuric acid and alkali treatment, substantially as described.

6. The process of obtaining purified distillates from Ohio or Lima oil, or in other words 25 from oil containing the impurities which are characteristic of the petroleum from the vicinity of Lima, Ohio, and which resist removal by the sulphuric acid and alkali or so-called Pennsylvania treatment, the said process consisting in distilling such oil mainly by heat 30 imparted thereto by conduction from metal surfaces, carrying on the distillation at a low temperature with the aid of an exhaustion of the vapors, and during such distillation subjecting the said impurities to the action of air 35 in volume sufficient to effect a substantial conversion thereof as indicated by a loss of the characteristic malodor of the aforesaid oil; substantially as described. 40

In testimony whereof I affix my signature in presence of two witnesses.

FRANCIS X. BYERLEY.

Witnesses:

SAMUEL A. BYERLEY,
IRA L. BERRY.