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G. O. GILMER.

PROCESS OF REFINING TURPENTINE.

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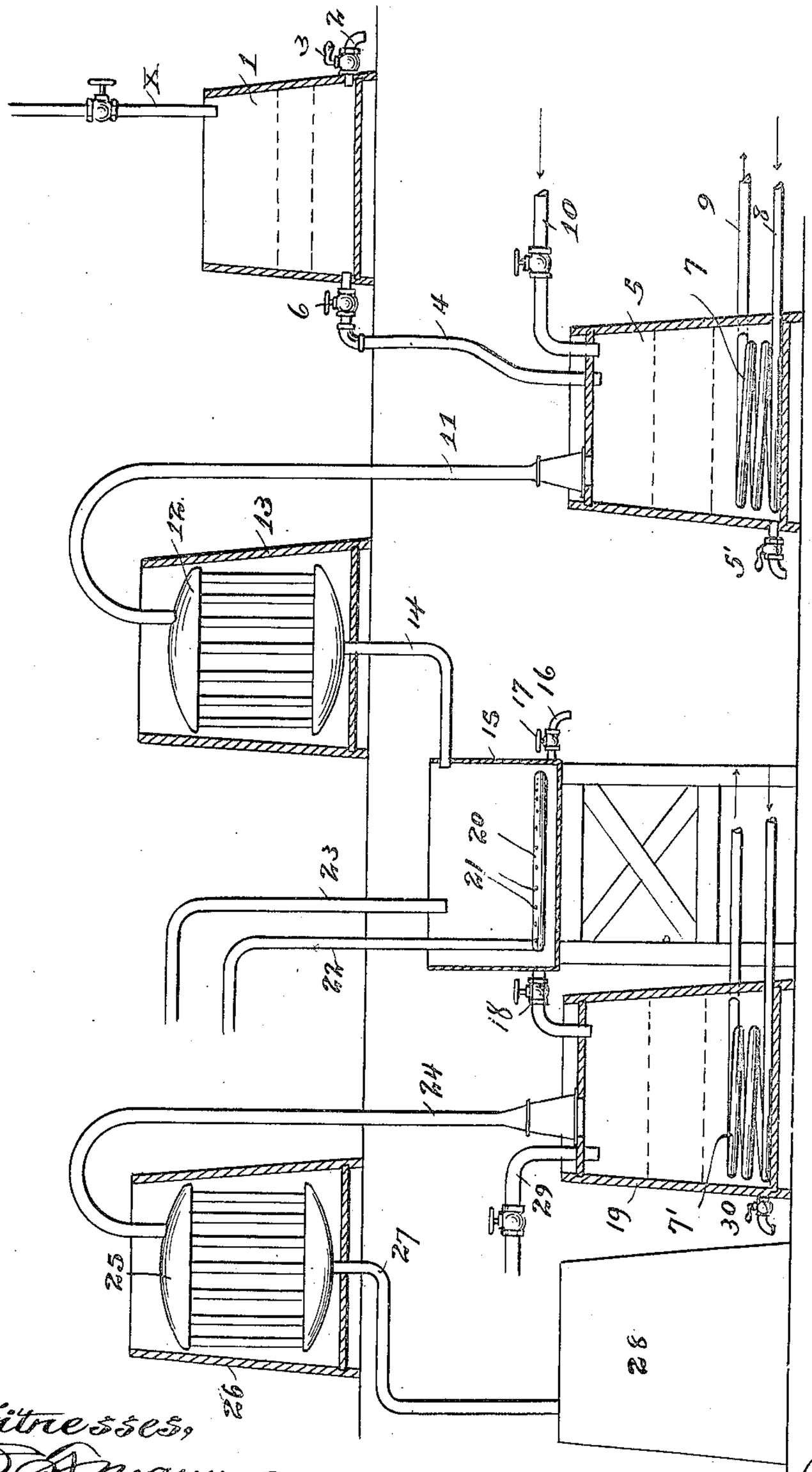

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UNITED STATES PATENT OFFICE.

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PROCESS OF REFINING TURPENTINE.

No. 813,088. Specification of Letters Patent. Patented Feb. 20, 1906.

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To all whom it may concern:

Be it known that I, GEORGE O. GILMER, a resident of New Orleans, in the parish of Orleans and State of Louisiana, have invented a certain new, useful, and Improved Process of Refining Turpentine, of which the following is a specification.

This invention relates to an improved process of refining turpentine; and it has for its salient objects to provide a process by which the crude turpentine is subjected to a series of successive redistillations and treatments which result in rendering the final product practically chemically pure and having improved characteristics and quality, to provide a process which may be carried out in a practically continuous manner and subject to such control during the carrying out of the process as to insure uniformly successful results, and in general to provide an improved process of the character referred to.

The turpentine to which my present improvement relates is obtained from the dry distillation of pine-wood in a closed retort, the process of distillation being such that the turpentine-vapors are expelled in advance of the driving off of the tarry and creosotic vapors to such an extent that the resulting turpentine product is impregnated to only a slight extent with such creosotic and tarry vapors and therefore is to be distinguished from previous patented processes wherein the material under treatment is pine-oil or tar-oil containing practically the whole of the tarry and creosotic ingredients. My ultimate purpose is to produce a chemically-pure turpentine from which the last vestige or remnant of creosote and other foreign ingredients is eliminated. I therefore begin with a product which may be described as a "crude turpentine," which is a thin liquid, about fifty per. cent of the volume being pyroligneous acid and the remainder, or one-half of the total volume, is about ninety-four per cent. turpentine with from four to six per cent. of creosotic tar.

To the above ends the invention consists in the matters hereinafter described and more particularly pointed out in the appended claims, and the process will be more readily understood by a description thereof as carried out by the use of one suitable type of apparatus illustrated in the accompanying drawing and in which the single figure illustrates the construction and arrangement of an apparatus suitable for the purpose referred to.

The invention is well adapted to refining turpentine produced by the descriptive distillation of wood, which crude turpentine generally consists of approximately forty per cent. of turpentine, fifty per cent. of water, two per cent. of creosote, and eight per cent. of tar.

In carrying out my process the crude turpentine is run into a suitable vessel and allowed to settle, whereby the acid, which has a greater specific gravity than the turpentine, collects upon the bottom and is drawn off, thus reducing the total volume of the liquid under treatment about one-half. The crude turpentine thus separated form the acid is mixed with about fifty per cent. of pure water and distilled off at a temperature of approximately 335° Fahrenheit. The vapor of the water forms a vehicle for the heavier vapors of the turpentine, and the vapors of this distillation are condensed by means of a suitable condenser and the liquids again separated by decanting after settling. The turpentine procured by the first distillation is practically or almost entirely free from tar, but contains a slight percentage of creosote. The tar separated out by this first distillation is a thick heavy tar and is in that condition ready for the market. In order to rid the turpentine of the remaining percentage of creosote, (which in the present process is so small that it is not saved,) the turpentine is next treated to a thorough washing and aerating in the presence of lime-water. To accomplish this, the turpentine is mixed with lime-water of a specific gravity about 3° or 4° Baumé and in the proportion of one part of lime-water to two of turpentine. The mixture is then subjected to a thorough agitation and aeration by blowing air therethrough. The washing and aerating having been concluded, the mixture is allowed to again settle and decanted to separate off the lime-water. The turpentine is then a second time distilled and the vapors condensed, care being taken to distil slowly, and thus avoid injuring the product. The vapors of distil-

lation are again condensed, and the resulting product is a turpentine which is practically chemically pure and free from tar or creosote.

Describing now the process as carried out by the use of the apparatus shown in the drawing, 1 designates a primary settling-tank, into which the crude turpentine may be discharged from the retort or from other sources of supply. The tank 1 is conveniently provided with a drawing-off pipe 2, located at the bottom level of the tank and controlled by means of a valve 3, through which pipe pyroligneous acid produced in the distillation may be withdrawn after the settling is completed. 4 designates a second pipe, arranged to communicate with the bottom of the tank 1 and leading thence to a primary still 5, said pipe 4 being likewise controlled by means of a hand-valve 6. The primary still is located at a lower level than the settling-tank 1, so that after the acid has been drawn off from the bottom of the tank the turpentine may be permitted to flow by gravity from the tank to the still. The still 5 may be of any suitable construction, that shown herein consisting of a closed tank, within the bottom of which is arranged a heating-coil 7, through which is circulated steam, the steam entering through an inlet-pipe 8 and returning through an outlet-pipe 9, which pipes, it will be understood, connect with any suitable source of steam-supply. It is to be noted that the heating-coil 7 is located at the bottom of the tank, so that it does not extend into the body of the turpentine, which latter is mixed with water in the primary still and becomes, therefore, supernatant. An inlet-pipe 10 is arranged to communicate with the primary still and with any suitable source of pure water, whereby the necessary supply of water may be supplied to the turpentine. With the top of the primary still a vapor-pipe 11 communicates, which extends upwardly to and discharges within a condenser 12, submerged in an elevated tank 13, said condenser 12 being of a usual and well-understood type, and therefore requiring no particular description. From the bottom of the condenser an outlet-pipe 14 leads to a tank 15, wherein the turpentine is subjected to a washing in the presence of lime-water, and it will therefore be designated the "neutralizing-tank." This tank is conveniently an ordinary open tank or tub provided at its bottom level with a drawing-off spout or pipe 16, controlled by a valve 17, and also with an outlet-pipe 18, which leads to and communicates with the secondary still 19, which may be identical in structure with the primary still 5. The neutralizing-tank is also provided with an air-distributing coil or pipe 20 located in its bottom and provided with a series of jet-openings 21, through which air may be blown into the liquid contained within the tank. With the aerating-coil 20 is connected an air-pipe 22, which communicates with any suitable source of air under pressure—as, for example, with an ordinary blower or air-pump. (Not shown). A supply-pipe 23 is also arranged to discharge into the neutralizing-tank, which supply-pipe communicates with any suitable source of supply of lime-water.

24 designates a vapor-pipe communicating with the secondary still and leading to a condenser 25, which may be identical with the condenser 12, hereinbefore referred to, and is similarly submerged in an elevated tank 26. From the bottom of the condenser 25 an outlet-pipe 27 leads to any suitable point of discharge—as, for example, to a storage-tank 28.

A suitable quantity of the liquid, consisting of pyroligneous acid and crude turpentine, having been charged into the settling-tank 1—as, for example, through the supply-pipe X—the mixture is allowed to stand until the pyroligneous acid and turpentine separate, the latter by reason of its higher specific gravity rising and floating upon the body of acid. After this separation has taken place the acid is then drawn off through the outlet-pipe 2, after which this outlet is closed and the remaining turpentine is drawn off through the pipe 4 into the primary still. To the turpentine thus charged into the still is added about fifty per cent. of pure water, which may be admitted through the water-pipe 10, whereupon the steam is admitted to the heating-coil and the water and turpentine distilled off. For this distillation steam at a temperature of approximately 335° Fahrenheit may be advantageously employed. The vapors passing out through the pipe 11 are condensed in the condenser 12 and flow on through into the neutralizing-tank 15. The tar which remains as a residuum in the primary still is drawn off in any suitable manner—as, for example, through the outlet-pipe 5′. The mixture which flows into the neutralizing-tank after the primary distillation is of course nearly fifty per cent. water. This water is drawn off from the bottom through the pipe 16, and thereafter the lime-water is added to the remaining turpentine, and the washing and neutralizing step performed by blowing air through the aerating-coil 20 and allowing it to escape at the top of the tank, the air being forced in under considerable pressure, so as to thoroughly agitate the contents of the tank, and this aerating process being continued for a considerable length of time, usually for about one hour. The aerating step having been completed, the air is shut off and the contents of the tank allowed to thoroughly settle, whereupon the lime-water at the bottom of the tank is drawn off through the outlet-pipe 16 and the remaining turpentine drawn off into the secondary still through the pipe 18. The secondary

still is filled with pure water to a point somewhat above the upper surface of the heating-coil 7′ therein, so that the turpentine charged into the secondary still is supernatant upon the water and does not come into contact with the heating-coil. A supply-pipe 29 is conveniently provided for admitting the water to the secondary still, and this still is likewise provided with a draw-off cock 30, through which the residuum, principally water, may be discharged. The second distillation is accomplished by means of superheated steam at a temperature of about 335° Fahrenheit for a period of about eight hours in order to distil off four barrels of turpentine. With a much lower temperature the process is too slow, and with a much higher temperature the distillation would carry over coloring-matter so that the turpentine would be inferior. The vapors pass from a secondary still into the condenser 25 and the liquids from the latter to the storage-tank 28. The turpentine may be decanted off as required from the storage-tank.

The product of the process is a turpentine which is practically colorless, is of the highest efficiency as a drier, and is in all respects equal to the best quality of orchard turpentine. Furthermore, as hereinbefore stated, it is practically chemically pure.

While I have herein described the process as carried out by the use of a particular form and arrangement of apparatus, it will of course be understood that any suitable apparatus may be employed for the purpose, and, furthermore, the steps of carrying out the process may be to some extent varied, as indicated by the broader claims.

I claim—

1. An improved process of refining crude turpentine, produced by dry distillation of turpentine-bearing wood which consists in first allowing the crude distillate to settle and then separating the turpentine-oil from the liquids of greater specific gravity by decanting, next adding to the turpentine-oil a large proportion of pure water, and distilling the mixture at a temperature sufficient to vaporize both water and turpentine, condensing the vapors of distillation, and again separating the turpentine-oil from the liquids of greater specific gravity by settling and decanting, next neutralizing the acid impurities of the turpentine-oil by introducing gradually a liquid alkali and maintaining an agitation during such addition of the alkali, arresting the introduction of alkali and the agitation of the mixture when the neutralization has been completed, allowing the mixture to settle and separating again by decantation, and finally redistilling the turpentine-oil in the presence of water and condensing and separating the turpentine and water vapors.

2. The process of refining crude turpentine produced by the destructive distillation of pine which consists in first separating the turpentine from the pyroligneous acid by settling, next adding approximately fifty per cent. of pure water and distilling the mixture at a temperature above that at which the water is vaporized, condensing the vapors of distillation, and allowing the liquids to settle and separate, next decanting off the water and adding to the remaining turpentine lime-water, then subjecting the mixture of turpentine and lime-water to air and an agitating process, next decanting off the lime-water and subjecting the turpentine to a second distillation, and finally condensing the turpentine-vapor.

3. An improved process of refining turpentine, which consists in first separating the turpentine from the pyroligneous acid contained therein, next adding approximately fifty per cent. of pure water and distilling the mixture at a temperature above that at which the water is vaporized, condensing the vapors of distillation, and allowing the liquids to settle and separate, next decanting off the water and adding to the remaining turpentine lime-water, then subjecting the mixture of turpentine and lime-water to an aerating and agitating process, next decanting off the lime-water, and then distilling the turpentine at a temperature of approximately 335° Fahrenheit, by means of heat transmitted to the turpentine from a body of water upon which the turpentine is supernatant and finally condensing the turpentine-vapor.

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