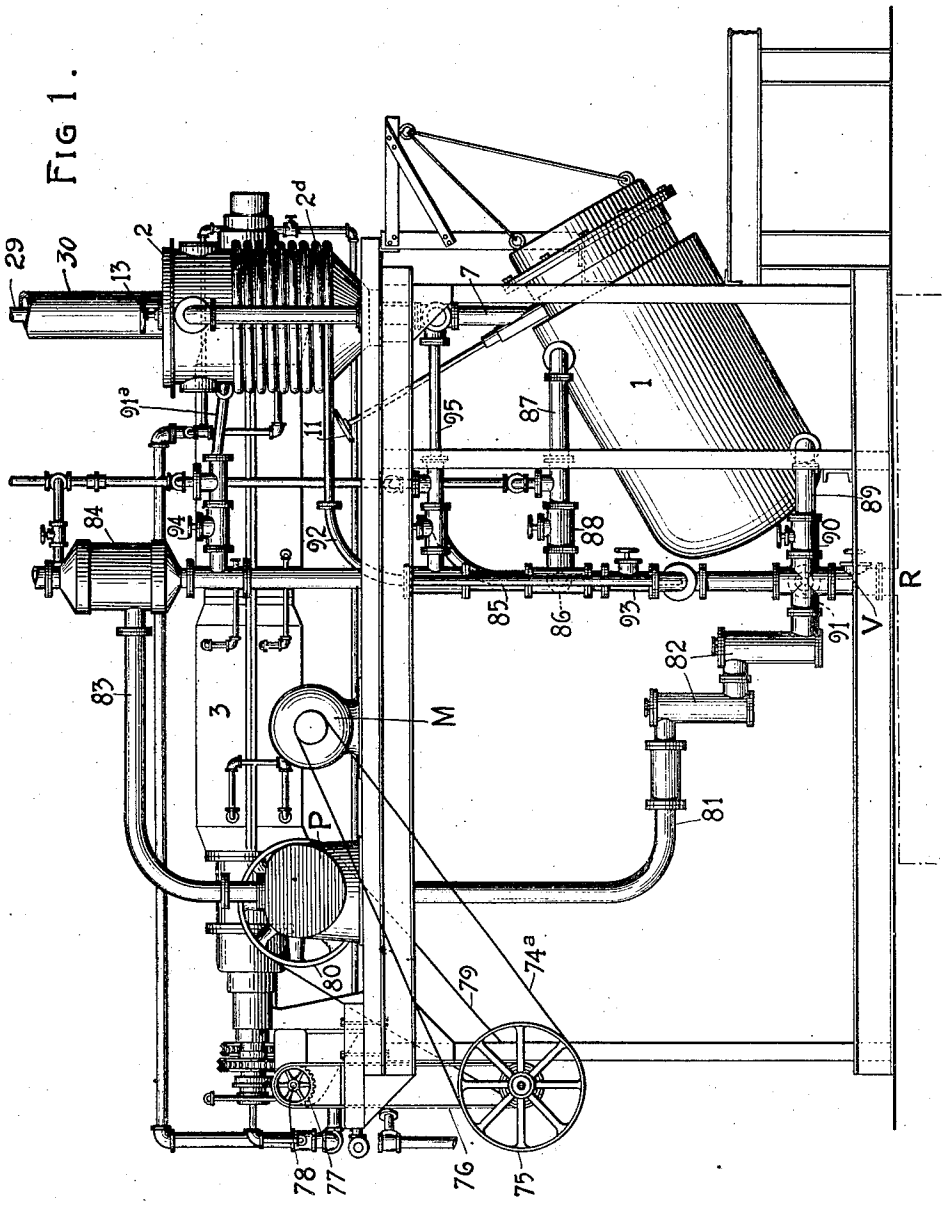
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J. E. CRANE ET AL

METHOD AND APPARATUS FOR PURIFYING CAMPHOR

Filed March 23, 1918 5 Sheets-Sheet 1

FIG 1.



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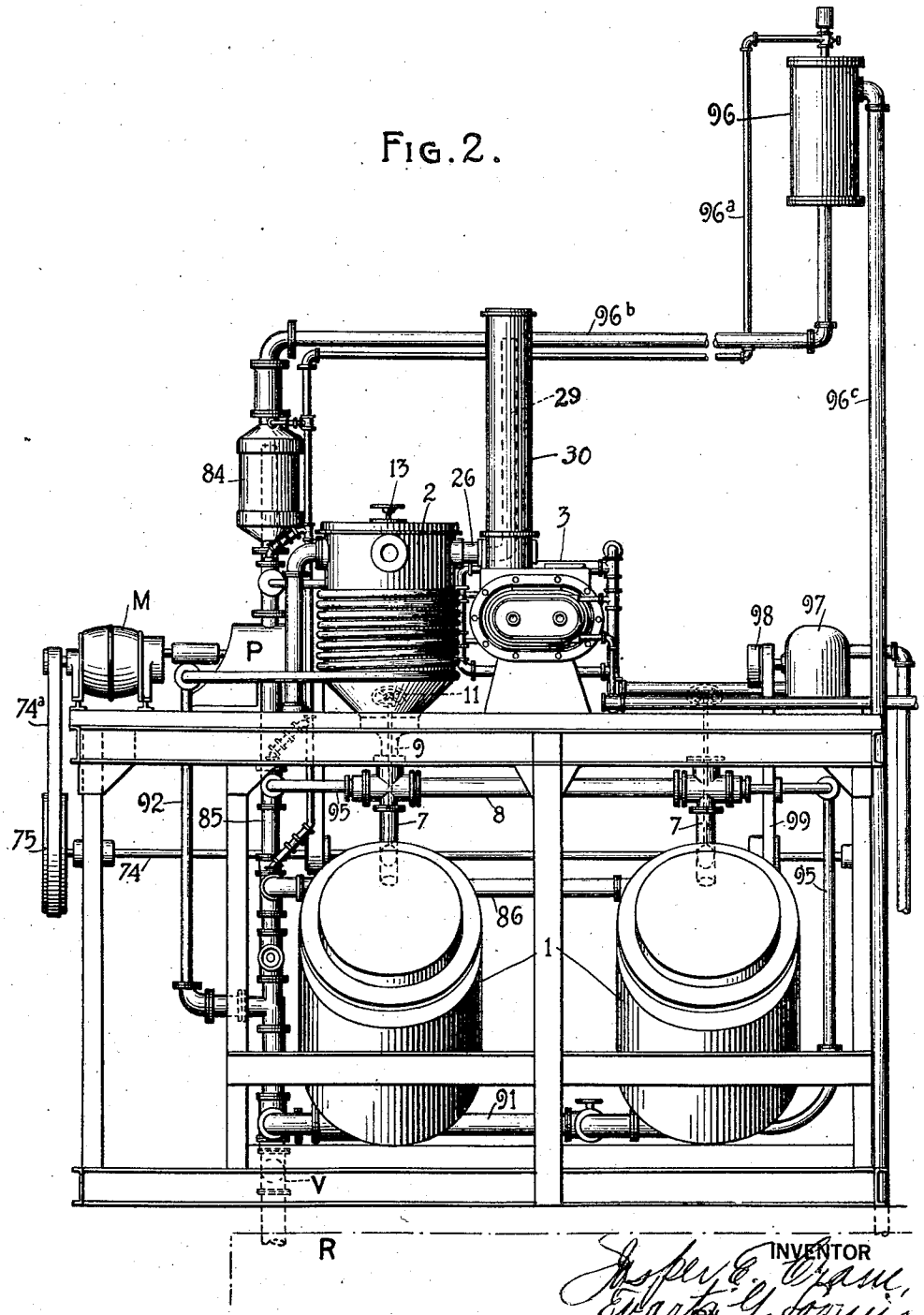
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METHOD AND APPARATUS FOR PURIFYING CAMPHOR

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FIG. 2.



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METHOD AND APPARATUS FOR PURIFYING CAMPHOR

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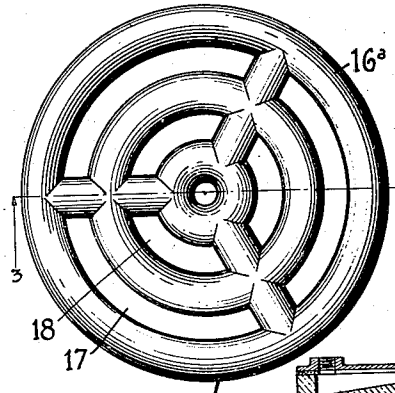


FIG. 3.

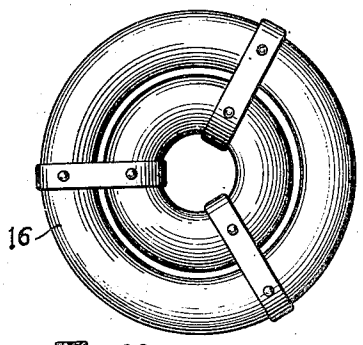
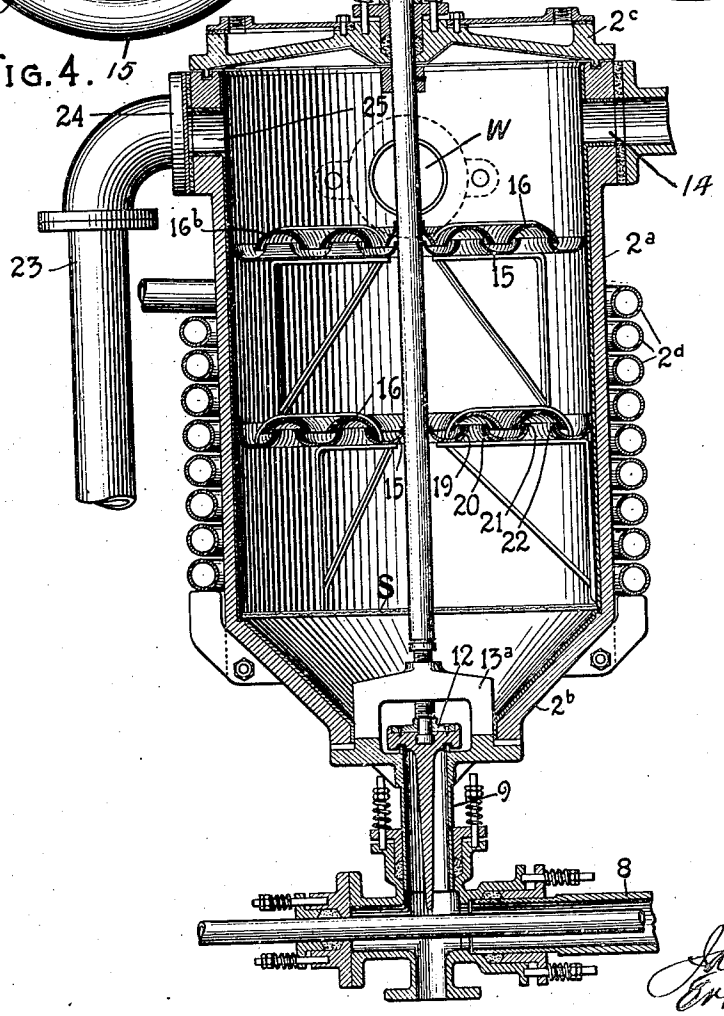


FIG. 4.

FIG. 5.



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J. E. CRANE ET AL

METHOD AND APPARATUS FOR PURIFYING CAMPHOR

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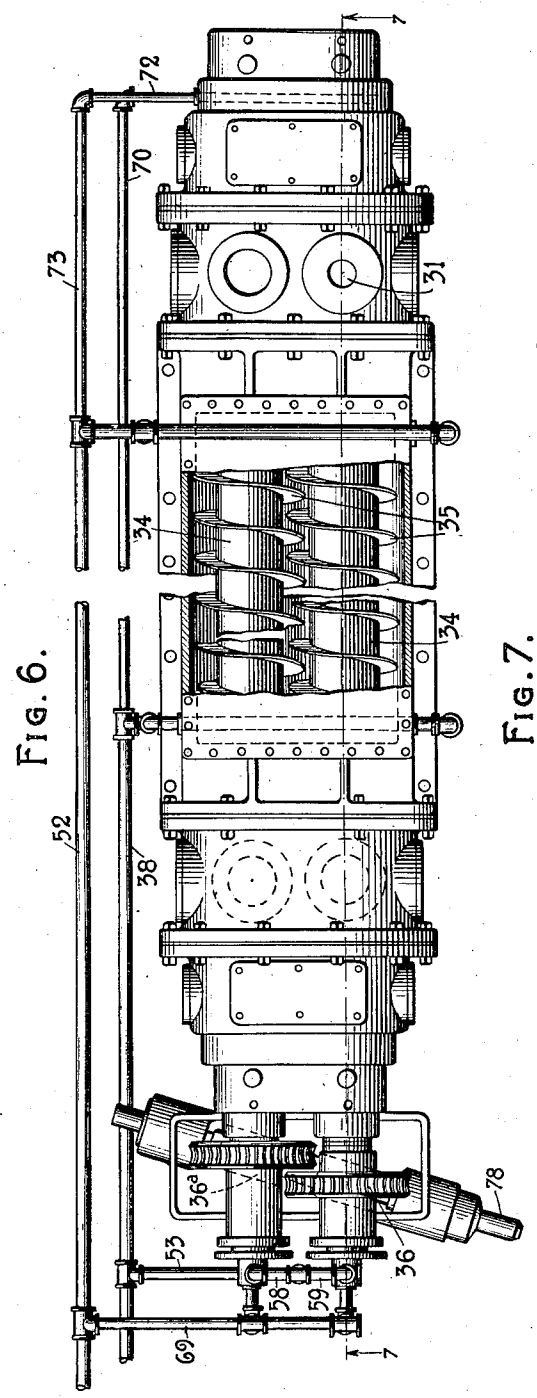


FIG. 6.

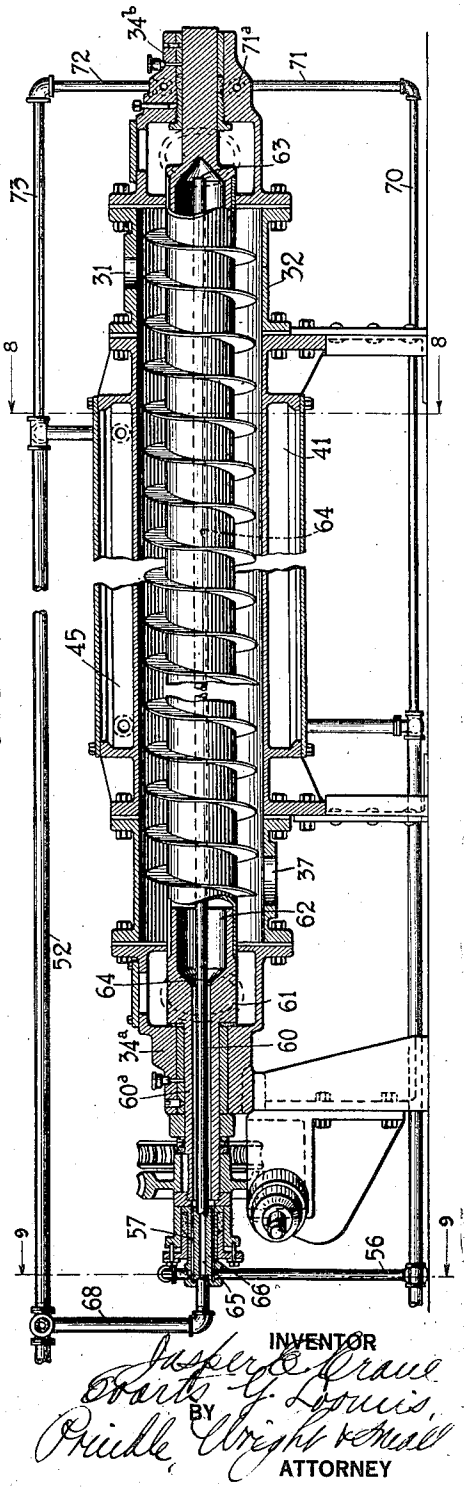


FIG. 7.

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METHOD AND APPARATUS FOR PURIFYING CAMPHOR

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FIG. 11.

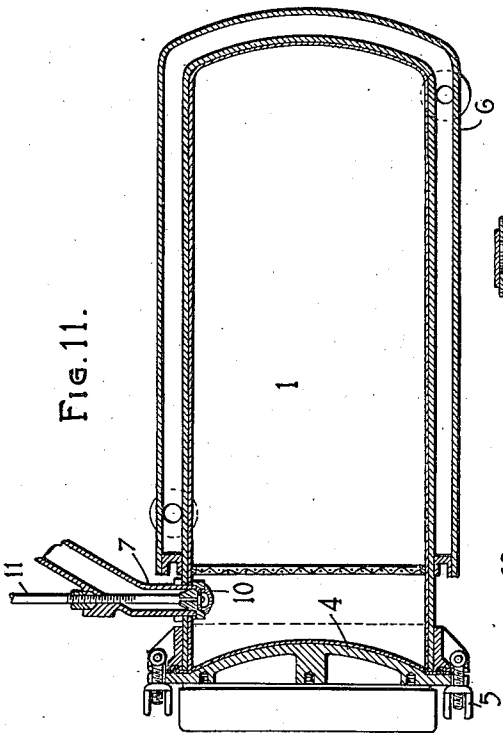


FIG. 10.

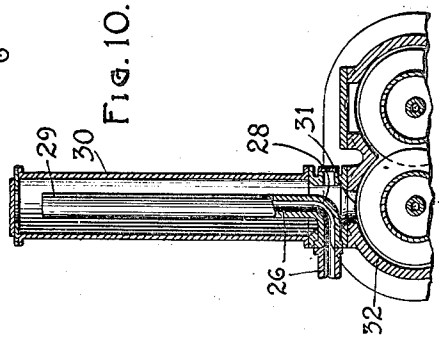


FIG. 9.

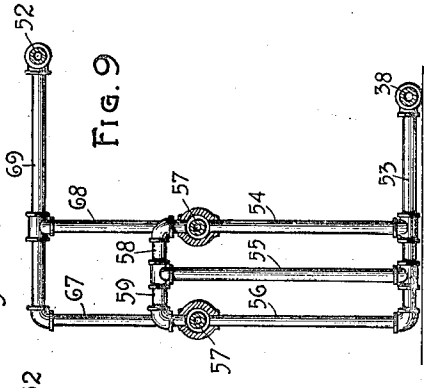


FIG. 12.

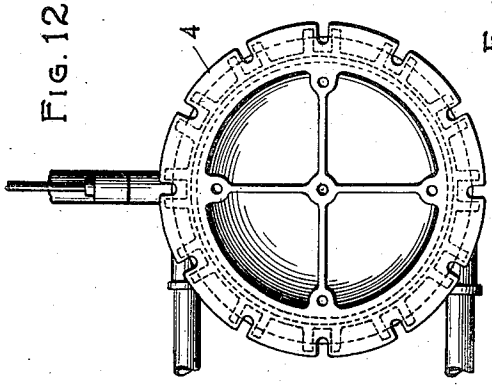
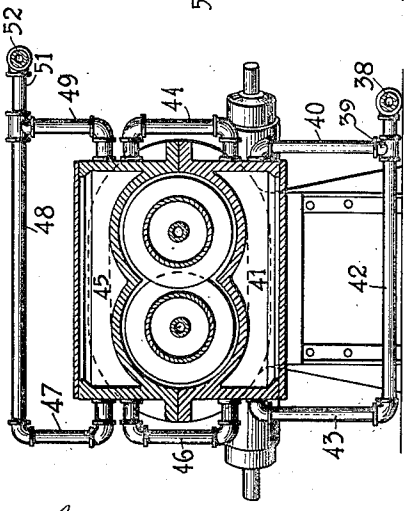


FIG. 8.



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BY

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Patented Sept. 18, 1923. 1,468,377

UNITED STATES PATENT OFFICE.

JASPER E. CRANE AND EVARTS G. LOOMIS, OF NEWARK, NEW JERSEY, ASSIGNORS TO E. I. DU PONT DE NEMOURS AND COMPANY, OF WILMINGTON, DELAWARE, A CORPORATION OF DELAWARE.

METHOD AND APPARATUS FOR PURIFYING CAMPHOR.

Application filed March 23, 1918. Serial No. 224,134.

To all whom it may concern:

Be it known that JASPER E. CRANE and EVARTS G. LOOMIS, of Newark, in the county of Essex and in the State of New Jersey, have invented a certain new and useful Improvement in Methods and Apparatus for Purifying Camphor, and do hereby declare that the following is a full, clear, and exact description thereof.

The invention has for an object to provide a method of treating camphor, so as to remove objectionable impurities from the same.

Another object of the invention is to provide an apparatus in which such process may be carried out to purify large quantities of camphor expeditiously and at low cost.

Further objects and advantages if the invention will appear from the detailed description thereof contained hereinafter, in which we have disclosed a specific form of apparatus and method of operation, which apparatus and method of operation are, however, to be considered merely as illustrative of the principle of the invention. In the drawings:—

Fig. 1 is a side elevation of an apparatus by means of which our invention may be carried out;

Fig. 2 is an end elevation of the same.

Fig. 3 is a vertical section of the purifier shown in Figs. 1 and 2.

Figs. 4 and 5 are plan views respectively of the pans and baffle plates preferably used in the purifier shown.

Fig. 6 is a plan view partially in section of the condenser shown in Figs. 1 and 2.

Fig. 7 is a section on line 7—7 of Fig. 6 looking in the direction of the arrows.

Fig. 8 is a section on line 8—8 of Fig. 7.

Fig. 9 is a section on line 9—9 of Fig. 7.

Fig. 10 is a vertical section showing in detail a preliminary cooler which may be used in connection with the condenser; and

Figs. 11 and 12 are respectively a longitudinal section and end view of a preferred form of baking chamber.

In the drawings, 1 represents a preferred form of baking chamber, of which any desired number may be employed in the apparatus, which chambers are preferably supported in inclined position by any suitable framework, as shown in Figs. 1 and 2. Baking chamber 1 (Fig. 11) may be made of steel or other suitable metal, and may be lined with any suitable material not subject to attack by camphor. To permit charging of the chamber a cover 4 is provided, held in place in any suitable manner as by wing nuts 5. The baking chamber is heated suitably, for example, by means of a jacket 6, through which heated oil from a circulating system later to be described, is passed. An outlet such as pipe 7 leads from chamber 1, such outlet being shown as controlled by valve 10 located therein and provided with an operating handle 11.

The outlet pipes 7 from the several baking chambers 1 are shown as connected to a header 8 which preferably leads, through pipe 9 to purifier 2, an element of especial importance to certain aspects of the present invention. The passageway through pipe 9 to purifier 2 is controlled by a valve 12, (Fig. 3), having an operating handle 13, and arms 13^a adapted to remove solidified camphor from the bottom of the purifier when the valve is rotated. The purifier 2 may be of any desired shape, but preferably consists of a cylindrical vessel 2^a, having a conical bottom 2^b, and a removable top cover 2^c to permit access to the same. One or more pans such as 15 are supported in the vessel 2^c between inlet pipe 9 and outlet pipe 14 thereof, such pans providing a plurality of troughs 16^a, (Fig. 4) separated by spaces 17 and 18. Baffle plates 16 are located above the pans 15 and are shown as having inverted troughs 16^b extending into troughs 16^a, and the latter are filled with a liquid such as camphor, through which any gases or vapors necessarily must pass in being led through the purifier. If desired, a further outlet opening 25 and pipe 23 may be provided, but the passage through such pipe is closed by disc 24, unless it be desired to divert the purified gases or vapors from the remainder of the apparatus. The purifier 2 is maintained at proper temperature, for instance by a coil of pipes 2^d, which are preferably included in a heated oil circulating system later to be described. A mica window W may also be provided for convenience in watching the operation in the purifier.

As shown, a pipe 26 leads from outlet opening 14, and preferably extends, as at

29, (Fig. 10), within the interior of a preliminary cooler 30, which cooler may be cooled by the air, or artificially if desired.

Preliminary cooler 30 is shown as mounted upon a condenser 3, a passageway 31 being provided between the two members. As shown particularly in Figs. 6 and 7 the condenser 3 perferably consists of an elongated casing 32 within which may rotate two screw conveyors 34, supported by means such as bearings 34^a and 34^b in the casing. The spiral blades 35 of the conveyors are preferably located in staggered relation to each other, such blades substantially filling the space within the casing 32, as shown in Fig. 8, and the two conveyors 34 are rotated in the embodiment shown, by means of worm gears 36 which engage a worm 36^a upon shaft 78. The outlet of the condenser 3 to which the camphor is advanced is shown at 37.

We have provided the following cooling system for the condenser, the details of which, however, may be widely varied. A pipe 38 containing cooling water, is connected by means such as pipes 40, 42 and 43 (Fig. 8), with a water jacket 41 located beneath the condenser casing 32. A similar water jacket 45 is shown as provided above such casing, and connected to jacket 41 through pipes 44 and 46; from jacket 45 the cooling water is led through pipes 47, 48, 49 and 51, to an exhaust pipe 52. The interiors of screw conveyors 34 are supplied with cooling water from pipe 38, by means such as pipe 53, leading through pipes 54, 55, 56, 58 and 59, to receiving chambers 57 such as are shown respectively adjacent the bearings 34^a of the screw conveyors 34. From chamber 57 the cooling water preferably passes through an annular space 60 in a neck 60^a of the screw conveyor 34, and thence to the hollow interior 62 of the body portion of the screw. The water thus flows the full length of the interior of such body portion, and back through the end 63 of a pipe 64, which serves to conduct the water to a fixed pipe 65, from which it is conducted to exhaust pipe 52, by means of pipes 67, 68 and 69 (Fig. 9). In order to cool bearings 34^b, we have provided means such as pipes 70 and 71 which lead from pipe 38 to a chamber 71^a within such bearing, and pipes 72 and 73 which are connected to exhaust pipe 52.

The means by which baking chambers 1 and purifier 2 are heated, is not essential to the invention, but such heating may be conveniently brought about by means of a heated oil circulating system as follows:—A reservoir R is provided, which is connected by pipe 81, which leads through electric heaters 82, to an oil pump P, a valve V being provided so that the oil supply in the system can be shut off from the reservoir if desired. From the pump P the oil is preferably delivered to a receiving chamber 84, from which it passes through suitable piping to the various apparatus to be heated. Piping 85 preferably leads from chamber 84, and is connected to the baking chambers 1 by means of a pipe 86 and branch pipes 87, leading respectively to the different baking chambers 1. Return pipes 89 may extend from the several baking chambers 1 to the pipe 91 which latter is shown as connected to pipe 81 so as to lead the oil back to pump P. Valves 88 and 90, located respectively in pipes 87 and 89 control the flow of oil to the different baking chambers. Heating oil is also shown as supplied to the purifier 2 from pipe 85 by means of a pipe 91^a, leading through coil 2^d to a pipe 92, which latter is shown as connected to pipe 85 on the low pressure side of a valve 93 located therein. Flow of oil to the purifier is controlled by means such as a valve 94 located in pipe 91^a. Header 8 is shown as also heated by pipe 95 extending therethrough from pipe 85, and leading back to pipe 91.

In order that there will always be sufficient pressure to force the oil through the heating system, and also in order that overflow of the oil may be provided for in case the amounts supplied by the pump should be more than can be taken care of upon the shutting off of the valves, in the embodiment shown a standpipe 96 (Fig. 2) is provided, which is connected by pipe 96^a to the pipe 85, and by pipe 96^b to the receiving chamber 84, and any over-flow to such standpipe is lead back to reservoir R through pipe 96^c.

By means of the circulating system, it will be obvious that any desired amount of heated oil may be supplied to the different apparatus to be heated, or the oil alone may be heated by opening valve 93 which brings about a "short circuit" of the circulating system. Also if desired, pump P may be operated at such speed that oil is continuously forced into the reservoir and pumped therefrom, thus making the reservoir a part of the circulating system.

Power for the pump P may be supplied from motor M, which drives a shaft 74 by means of a belt 74^a and pulley 75, shaft 74 driving the pump through belt 79 and pulley 80. Worm shaft 78, which operates the conveyor, may also be driven from shaft 74 by means of belt 76 and pulley 77. If cooling water under pressure is not otherwise readily available, a water pump 98 may also be driven from shaft 74 through pulley 98 and belt 99.

In purifying camphor according to the method of operation which is preferably followed with the above described apparatus, crude camphor is fed into baking chambers 1 through covers 4, and the chambers closed.

Sufficient heat is applied, by means of the oil circulating system above described, to vaporize the crude camphor, with valves 10 closed. The heating in confinement is preferably continued until substantially all the camphor oils and resinous products contained in the crude camphor are decomposed, and converted into relatively non-volatile substances, or, at least, into substances having different volatility from camphor. At atmospheric pressure, camphor melts at approximately 350° F., and boils at about 400° F., and a temperature of about 500° F., will convert the camphor into vapor form without decomposing it or altering it chemically, and such temperature if maintained for approximately one hour is sufficient to decompose the above mentioned impurities. If desired, the water may be removed at this time by inserting into the baking chamber such material as lime. It will be obvious that any desired number of the baking chambers may be utilized in performing the heating operation with the valve 10 closed, while other baking chambers may be either inoperative or performing the subsequent distilling operation.

After the organic impurities have been decomposed, and the camphor vaporized as above described, valves 10 and 12 are opened, so that the camphor vapors or fumes pass through header 8, pipe 9 into purifier 2. In this latter member the fumes are compelled to pass through a body of liquid, such as molten camphor, as above described contained in troughs 16ª; and this scrubbing operation removes such foreign matter, as dirt or dust etc., which may have become mechanically mixed with the camphor fumes. The organic impurities having been left behind and thereby removed in the baking chamber, and the camphor also having been purified as above described in the purifier 2, substantially pure camphor fumes will pass through outlet 14 and pipe 26, which fumes are in condition to be condensed and collected.

If the preliminary cooler 30 is employed the fumes will next be led through it, and their temperature reduced thereby, after which they pass through opening 31 to the interior of condenser casing 32. At this point it is probable that the camphor might be observed both in vapor and liquid form, and owing to the cooling of the condenser casing, condensation of the camphor upon the walls thereof soon will take place. Such condensation is assisted by the provision of the spiral blades which form an elongated passageway through which any fumes must pass in being led through the casing, whereby the blades hold the camphor back, and cause it to condense readily. As the camphor solidifies the spiral blades scrape it away from the cooling surfaces, and at the same time convey it continuously through casing 32 until it is finally delivered at outlet opening 37. The spiral blades of the conveyors 34, being staggered, rotate in such a manner that the blades of one conveyor will always clear the blades of the other, so that each blade acts to scrape out the spaces between the turns of the other blade. In this manner the camphor fumes are changed from a gaseous to a solid state, during all of which time the camphor is being transferred from the entrance end of the condenser toward the delivery end thereof.

While we have described a specific form of our invention, it is obvious that many changes will be made, which will still be within the spirit thereof as defined in the following claims.

We claim:

1. The method of purifying camphor which comprises vaporizing camphor from the crude product, passing the resulting fumes through liquid camphor, and cooling the fumes to condensation.

2. The method of purifying camphor which comprises passing camphor fumes through liquid camphor, cooling said fumes to condensation, and removing the solid camphor from the condensing chamber as it is condensed therein.

3. The method of purifying camphor which comprises confining and heating the crude product to a temperature sufficient to vaporize the camphor from same and decompose organic impurities, and cooling the fumes to condensation.

4. The method of purifying camphor which comprises confining and heating the crude product to a temperature sufficient to vaporize the camphor from same and decompose organic impurities, scrubbing the fumes by passing them through a liquid, and cooling the fumes to condensation.

5. The method of purifying camphor which comprises confining and heating the crude product to a temperature sufficient to vaporize the camphor from same and decompose organic impurities, passing the fumes through liquid camphor, and cooling the said fumes to condensation.

6. The method of purifying camphor which comprises confining and heating the crude camphor to a vaporizing temperature and maintaining the temperature long enough to decompose the impurities in the crude camphor, scrubbing the camphor fumes through liquid camphor, and condensing them while continously removing the condensate.

In testimony that they claim the foregoing they have hereunto set their hands.

JASPER E. CRANE.
EVARTS G. LOOMIS.

Witnesses as to Jasper E. Crane:
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ADE SCHWEINITZ.