C. E. MOLESWORTH.

DISTILLING APPARATUS.

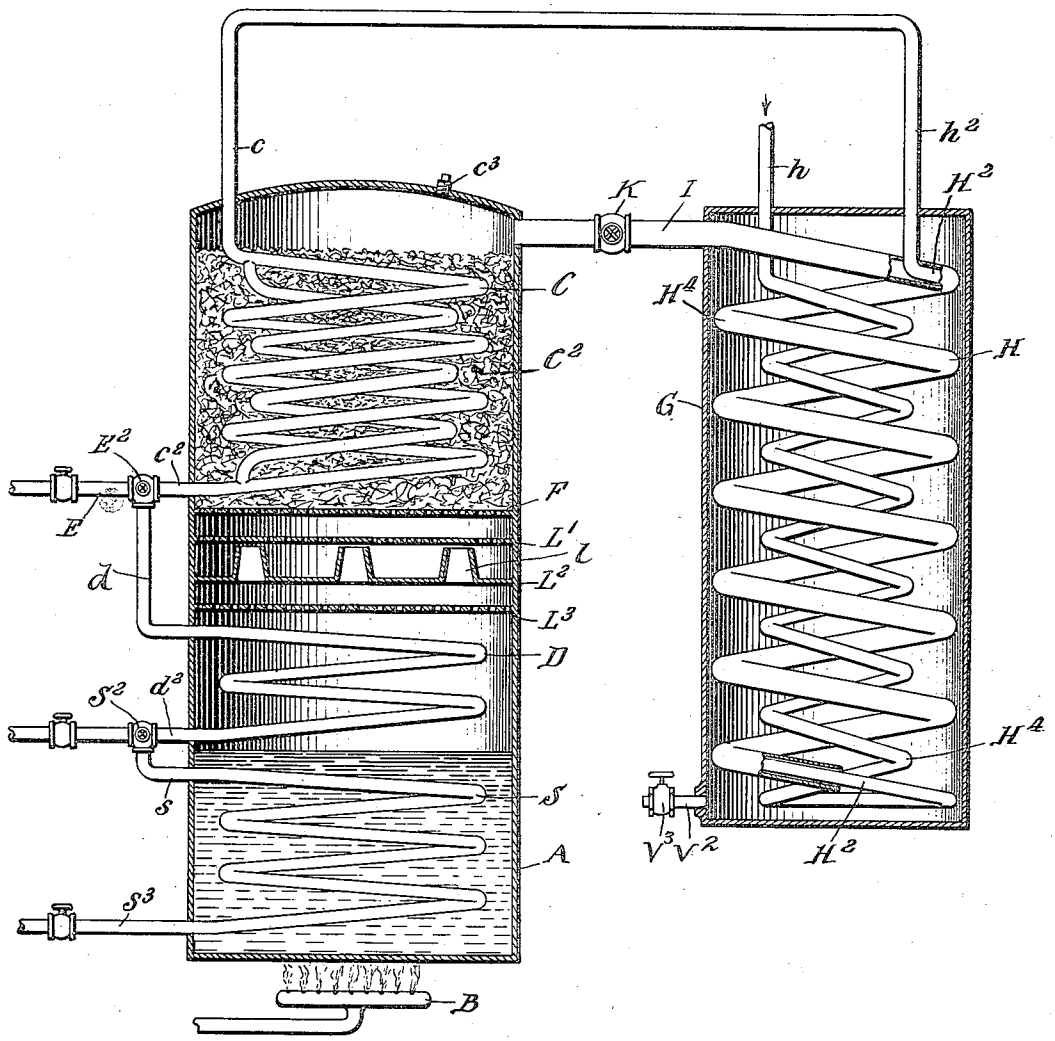
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1,086,684.

Patented Feb. 10, 1914.

2 SHEETS—SHEET 1.

Fig. 1.



Witnesses

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Inventor

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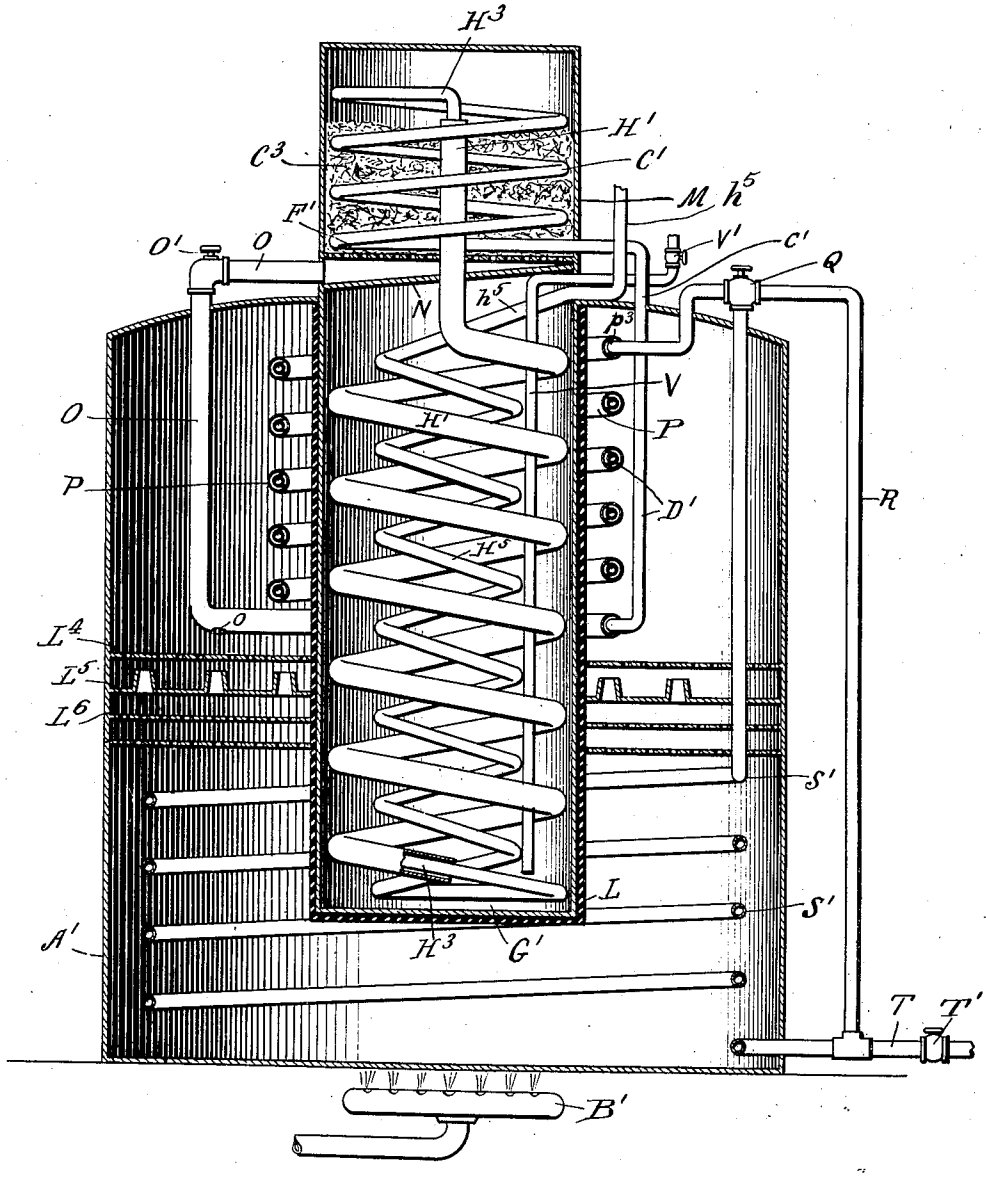
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2 SHEETS—SHEET 2.

Fig. 2.



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UNITED STATES PATENT OFFICE.

CLYDE E. MOLESWORTH, OF PORTLAND, OREGON, ASSIGNOR TO THE REFRIGERATION CORPORATION, OF NEW YORK, N. Y., A CORPORATION OF NEW YORK.

DISTILLING APPARATUS.

1,086,684. Specification of Letters Patent. Patented Feb. 10, 1914.

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To all whom it may concern:

Be it known that I, CLYDE E. MOLESWORTH, a citizen of the United States, residing at Portland, in the county of Multnomah and State of Oregon, have invented certain new and useful Improvements in Distilling Apparatus, of which the following is a specification.

My invention relates to improvements in distilling apparatus for the separation of two liquids by removing one of them from the other by distillation.

The object of my invention is to provide a distilling apparatus especially adapted for the separation by distillation of two liquids, one of which has a boiling point lower than ordinary atmospheric temperatures under normal atmospheric pressures.

With this general object in view my invention consists in the features, details of construction and combination of parts which will first be described in connection with the accompanying drawings and then pointed out in the claims.

In the drawings Figure 1 is a diagrammatic view of one embodiment of my invention; Fig. 2 is a similar view illustrating another embodiment of my invention.

Referring to the drawings and particularly to Fig. 1, A indicates a still arranged to contain the mixed liquids to be separated. This still is arranged to be heated by any desired means as, for example, the gas heater B illustrated in the drawings. In the head of the still, in the present example, is arranged a cooling coil indicated at C into which leads an inlet pipe c and is arranged to discharge outside of the still, as at c^2. In addition to this the still is provided with a second coil indicated at D and arranged below the coil C, the second coil D having an inlet d and being arranged to discharge outside the still as indicated at d^2. A waste pipe E and a three-way cock E^2 are provided in connection with the outlet c^2 and the inlet d of the respective coils C and D so that by turning this three-way cock E^2 the outlet of the coil C may be connected with the inlet of the coil D or in another position the coil D may be cut off from communication with the coil C and the outlet c^2 of said coil C may be connected with the waste pipe E.

At F the still is provided with a diaphragm of any suitable construction as, for example, a perforated diaphragm, above which is placed a packing C^2 of suitable sub-divided material which will be chemically permanent in the presence of the liquids being treated. I have found that a mineral wool made from glass is especially adapted for my purpose. This mineral wool is packed around the cooling coil C in such a manner as to form a porous mass having a very large surface.

At G is indicated a receptacle containing three coils, H, H^2 and H^4. The pipe forming the coil H^2 is arranged within the pipe forming the coil H, the diameter of the two pipes being such that a suitable free space is left within the pipe of the coil H for the passage of the vapor while the pipe H^2 serves to conduct a suitable cooling liquid. The coil H^4 is preferably arranged concentric with the coil H and, in the present example, within the convolutions of said coil H. The lower ends of the coils H^4 and H^2 are connected with each other and the upper end of the coil H^4 is provided with an inlet h for the cooling fluid while the coil H^2 is provided at its upper end with an outlet h^2 passing through the wall of the coil H and through the wall of the receptacle G, a gas tight joint being made at each of said walls by suitable methods as, for example, by welding processes. The outlet h^2 is connected with the inlet c of the coil C. The head of the still A is connected with the upper end of the coil H in a suitable manner as by means of a pipe I, which is provided with a valve K.

In the apparatus thus far described the operation is as follows: The mixed liquids to be separated by distillation are filled into the still A to a level somewhat below the bottom of the coil D, a suitable filling opening being provided for that purpose as indicated at c^3, this opening being closed by any suitable means as, for example, by a plug. A cooling fluid such, for example, as water, is caused to enter the inlet h whence it flows first downward through the coil H^4, thence upward through the coil H^2, thence to and through the inlet pipe c to the upper cooling coil C, whence it flows through said coil and either discharges through the waste pipe E or, if the stop-cock is turned in the proper manner, it will flow downward through the coil D and discharge through the outlet d^2 of said coil.

Heat being applied to the still, as by lighting the burner B, vapors are driven off from the mixed liquid. In the beginning of the operation these vapors consist principally of vapors of the lowest boiling liquid, but a limited amount of vapors and of particles of the higher boiling liquid accompany the vapor of the more volatile one, being carried up by mechanical action, or entrainment, etc. As the arising vapor passes through the space cooled by the cooling coil D the temperature of such vapor is somewhat lowered and in its further passage upward it is compelled to traverse the perforations in the partition F and escape through the various pores or openings in the packing C^2 which compels the said vapors to come into intimate contact with the particles of the packing which are kept at adjusted temperature or cooled by the cooling liquid passing downward through the coil C. In this way the temperature of the vapor is lowered and made uniform and the particles of the high boiling liquid are forced into contact with and carried off by liquid on the surface of the particles of said packing material C^2. This liquid is retained in part on such particles of the packing material in the form of a film, by surface tension, the excess gradually dripping back to the still bottom. In this way the vapor of the low boiling liquid is freed from the high boiling liquid to the maximum extent and passes from the still through the pipe I to the upper end of the coil H. It passes thence down said coil around the coil H^2 and is gradually cooled on its downward path until finally it escapes from the coil H near the bottom of the receptacle G gradually accumulating in said receptacle G and creating a pressure therein. When the pressure of the said vapor is sufficiently high a condensation of said vapor (if the vapor be one not liquefying at the ordinary temperature and pressure) to a liquid form takes place and thereafter the vapor entering the pipe I and passing through the coil H condenses to liquid by the time it leaves the lower end of the coil H so that eventually a larger part, if not all, of the low boiling liquid is driven from the still charge and condensed in the receptacle G in a liquid form from which it may be withdrawn through any suitable discharge pipe, as for example the one indicated at V^2, controlled by a suitable valve V^3. If desired the controlling valve K can be closed on initially starting the still until a sufficient pressure has accumulated in the still, whereupon the valve may be opened and the vapor of the low boiling liquid permitted to escape through the coil H into the receptacle G where it will liquefy practically immediately, if the pressure is sufficiently high in relation to the temperature to which the said vapor is cooled by the cooling liquid in the coil H^2. It will be seen that the point of lowest temperature in this apparatus is practically at the bottom of the receptacle G and that this temperature will determine the pressure in the receptacle G which in turn determines the pressure in the still.

It is important that the cooling liquid flowing through the coil C in the still proper shall not be cold enough to condense the vapor of the low boiling liquid in the still but at the same time it should have a temperature only slightly above the point of condensation of said low boiling vapor that the packing material may be as efficient as possible in removing the vapors and entrainment of high boiling liquid. Since the cooling coil C in my apparatus receives its supply of cooling fluid from the outlet of the cooling coils in the receptacle G it will be clear that the cooling fluid entering said coil C will be at a temperature higher than that of the coldest part of the apparatus, owing to the flow of the cooling liquid upward in the coil H^2 counter to the flow of the incoming vapor in the coil H. As the temperature of the coldest part of the apparatus is at the point of condensation of the low boiling liquid, the cooling liquid, as it leaves the outlet h^2 of the coil H^2, is warmer than the said temperature of condensation of the low boiling liquid under the conditions then existing, because said cooling liquid absorbs heat on its way out and as this cooling liquid passes from the outlet pipe h^2 to the coil C, it follows that the temperature of the coil C must at all times be slightly above the point of condensation of the vapor of the low boiling liquid, thus insuring that at no time shall the temperature of the coil C fall to a point where it could cause any condensation whatever of said vapor of the low boiling liquid. Furthermore, as the temperature of the coil C determines the temperature of the sub-divided packing material in contact with it, owing to the conductivity of the surface film of high boiling liquid on the particles of such packing material, every part of the vapors arising from the still is compelled to contact with this surface film whose temperature can never be low enough to condense the vapors of the low boiling liquid but is always low enough to insure a complete deposition of the entrained high boiling liquid.

It will be seen that the sub-divided packing material offers a large surface area for catching the entrainment, and by virtue of the very minute sub-divisions of the arising vapor due to its being compelled to pass through the minute spaces or pores in this packing material, all parts of the vapor are brought into intimate contact with the parti-

cles of the packing material, thus making it impossible for any portion of the vapor to carry past the packing material any entrained liquid. Furthermore, as this packing material is cooled by the cooling coil C, to the lowest permissible temperature without causing condensation of the low boiling constituent, the vapor tension of the high boiling constituent is reduced to its minimum value, so that the vapors after passing the packing material contain but a relatively insignificant proportion of the high boiling constituent in vapor form. The result of this is that the work to be done by the condenser is thereby reduced and at the same time the condensate is as free as possible from the high boiling constituent. Moreover, by my apparatus, the temperature of the packing material is controlled, not directly by the conditions of operation of the still but by those of the condenser, which in turn are dependent upon the temperature of the cooling fluid. Hence, the result is an apparatus which is automatic in action as to the control of temperature by the cooling coil C, and thus avoids the necessity for the exact attention and control usually required in operating such stills. In addition to this there are many other advantages in an apparatus working at low pressures, for example, lower cost of construction, less danger of leaks, less weight and volume, all of which are obvious to those skilled in the art.

For convenience, the still A may be provided with an additional cooling coil in the liquid space, as indicated at S and the inlet s of this coil may be connected with the discharge outlet of the coil D by a three-way cock S^2, so that when it is desired to cool the liquid remaining in the still after the completion of the operation, the three-way cock S^2 may be turned so as to cause the cooling fluid from the coil D to pass through the coil S and thence outward through the discharge pipe s^3.

For the purpose of avoiding disadvantages which might arise through priming and foaming of the still charge I provide the still with three diaphragms indicated at L', L^2 and L^3 respectively, the two diaphragms L' and L^3 being perforated while the intermediate diaphragm L^2 is provided with openings having upward extending tubules as indicated at l. Owing to the presence of these diaphragms any foam due to the ebullition of the still-charge will be broken up in passing through the partitions and thereby the vapor separated from the liquid, which can drain back into the bottom of the still. This is an important feature in connection with the use of the cooled packing material.

Referring to Fig. 2 of the drawings, which shows diagrammatically a modification of my apparatus, A' indicates the still, G' is a receptacle arranged concentrically with the still and projecting within the same, the said receptacle being insulated by a suitable jacket of heat insulating material as indicated at L. Above the receptacle G' is located a container M separated from the receptacle G' by a partition N, the container having a gas inlet pipe O entering substantially on a level or slightly below the level of the upper surface of the partition N for a purpose hereinafter explained. The gas inlet pipe O is connected with the lower end of a coil P located in the head of the still, the upper end p of this coil being open to the interior of the still. Within the coil P is contained a cooling coil indicated at D' through which cooling coil flows the cooling fluid as hereinafter more specifically pointed out. The receptacle G' contains three coils H', H^3 and H^5. The pipe of the coil H' incloses the pipe of the coil H^3, the latter carrying the cooling fluid while the space between the two pipes of the two coils H' and H^3 conveys the gas to be cooled. The bottom ends of the coils H^3 and H^5 are connected. The upper ends of the coils H' and H^3 extend through the partition N and upward to the upper end of the container M at which point the coil H' is open to the interior of the container while the upper end of the coil H^3 connects with a cooling coil C' whose outlet c' connects with the inlet of the cooling coil D' in the still. The coil C' is surrounded by suitable sub-divided packing material C^3 as referred to more particularly in connection with the form of apparatus shown in Fig. 1, this packing material being supported on a suitable perforated diaphragm F' in the container.

The apparatus thus far described operates in substantially the same manner as the apparatus shown in Fig. 1 and briefly as follows: Heat being applied to the still from any suitable source, as from a burner B', an ebullition is caused in the liquids contained in the still. The arising vapor is cooled by the cooling coil D' indirectly at first and then directly when it enters the inlet p^3 of the coil P and passes downward through said coil in close contact with the cooling coil D'. The somewhat cooled vapor passes upward through the pipe O and enters the bottom of the container M from whence it passes through the diaphragm F' and through the pores of the packing material C^3 where any particles of the high boiling liquid will be caught by the sub-divided material and caused to drain back to the bottom of the container M while the vapor of the low boiling material passes to the upper part of the container and thence downward through the coil H' in close contact with the cooling coil H^3, said vapor being discharged into the receptacle G'. The high boiling liquid screened out of the vapor by the packing material C^3 flows back through the pipe

O and enters the still through a small hole *o* at the bottom of the pipe O. The cooling fluid enters the upper end of the coil H^5 through an inlet h^5 and passes downward toward the bottom of the receptacle G′ and thence upward through the coil H^3, thence downward through the coil C′ and finally reaches the bottom convolution of the coil D′ within the still, from whence it passes upward through said coil and then out. At Q is indicated a three-way cock arranged to connect the outlet of the coil D′ with a waste pipe R or with the inlet *s*′ of a coil S′ located within the liquid space of the still A′, the said coil S′ discharging outward into the waste pipe T. A suitable valve O′ is provided in the pipe O for the purpose of controlling the apparatus.

For the purpose of controlling the flow of cooling fluid, suitable valves may be supplied. However, as it is usual with an apparatus of this kind to have the various cooling coils supplied from a city water system or the like, I have found it a great advantage to put the controlling valve T′ for the cooling fluid on the outlet of the system rather than on the inlet for the reason that when the cooling fluid is shut off by such valve on the outlet pipe the pressure of the cooling fluid is acting on the interior of all of the coils, thus tending to counteract the pressure on the exterior of said coils.

In the modification of the apparatus shown in Fig. 2 the still may have diaphragms L^4, L^5 and L^6, whose function is similar to the corresponding diaphragms in the form of apparatus shown in Fig. 1.

The form of apparatus shown in Fig. 2 has a number of important advantages in addition to that of compactness. Probably the most essential advantage is that leakage or sweating through the walls of the receptacle G′ of the condensate under pressure in said receptacle will not result in an escape of said condensate into the atmosphere but merely into the still A′ where said condensate will expand and thereby be reduced in pressure or be absorbed in the high boiling liquid in said still. The still itself is, of course, only under a relatively high pressure during its operation whereas the receptacle G′ would be under pressure at all times except when emptied, which may be done through an outlet pipe V controlled by a valve V′. Furthermore, in case the receptacle G′ is filled with condensate and therefore under pressure, any great increase in temperature of the apparatus as, for instance, in case of fire, would cause a much greater increase in pressure in the receptacle G′ than in the still A′. If this accidental increase of pressure should cause any rupture of the receptacle G′, the contained condensate would escape into the still A′ and thereby the pressure would be materially reduced, first, because of the expansion of the condensate into the still and, second, because a large part of said condensate would be reabsorbed by the high boiling liquid in the still A′.

In practice it is not always possible to make the receptacle G′ of such high quality as to absolutely avoid the presence of some porous material and hence it is very generally the case with such apparatus that a certain amount of the condensate in the receptacle G′ will ooze through such porous spots in the walls of said receptacle. Usually this escape of material is so small as to be unnoticed but, at the same time with many fluids it may cause disagreeable odors around the apparatus. With the construction shown in Fig. 2, however, any such oozing of the condensate through the wall of the receptacle G′ merely results in a slight escape of the material back to the still where it is not lost but may be again driven back into the receptacle on the next operation of the still.

Another very important feature of the type of apparatus shown in Fig. 2 is that when the valve O′ in the vapor pipe from the still to the bottom of the container is closed, the vapor of the low boiling constituent in the container is shut off from the still, but has a passage way open to the condensate receptacle through the condenser worm, H′. Hence, when the source of heat is cut off from the still there is no possibility of a reabsorption of this vapor by the high boiling liquid in the still, as there would be in the apparatus shown in Fig. 1. This avoids the loss of a considerable portion of the distilled vapor which would otherwise occur.

My invention is particularly adapted for use in ammonia refrigerating systems, wherein the still is used for driving off the ammonia from the weak liquor, the said ammonia being condensed in the receptacle for condensate, from whence it may be withdrawn as needed and after expansion in the usual refrigerating coils may be returned to the still. In apparatus of this type constructed and operated by me, the cooling liquid was water from the usual city supply and the source of heat was gas burners supplied with the usual city gas.

Having thus fully described my invention, what I claim is:

1. In a distilling apparatus, the combination, with a still and means for heating the same, of a condensate receptacle, a condensing worm within said receptacle, a cooling coil telescoped in the worm and provided with an inlet for cooling fluid, a second cooling coil in the receptacle having its lower end connected with the lower end of the first cooling coil and having an outlet, a

sub-divided packing material arranged to be traversed by the vapors on their way to the worm, a third cooling coil arranged in said packing material, and connections whereby the cooling fluid from the outlet of the second cooling coil traverses the third cooling coil in a direction opposite to the flow of vapors through the packing material.

2. In a distilling apparatus, the combination, with the still, and a condenser, of a cooling coil, a mass of sub-divided packing material inclosing said cooling coil and arranged to be traversed by the vapors on their way to the condenser, and means for controlling the temperature of the cooling fluid in the said cooling coil by the temperature of the condenser.

3. In a distilling apparatus, the combination, with a still, and a condenser having a counter current cooling coil, of a container, a mass of inorganic fibrous material in said container, connections whereby the still vapors are compelled to pass through said fibrous material on their way to the condenser, a second cooling coil embedded in said mass of fibrous material, and connections whereby the cooling fluid from the condenser passes through the said second cooling coil in a general direction opposite to the flow of vapors through the fibrous material.

4. In a distilling apparatus, the combination, with a still, of a receptacle for condensate, a condensing worm within said receptacle and arranged to receive the still vapors at its upper end, a cooling coil telescoped in the worm and having an outlet at its upper end, and a second cooling coil within the receptacle and having an inlet at its upper end, the lower ends of both cooling coils being connected.

5. In a distilling apparatus, the combination, with a still, of a condensate receptacle located within the upper part of the still, a wall of insulating material surrounding the receptacle, condensing means located within the receptacle, connections whereby the still vapors are condensed and the condensate discharged into the receptacle, a container located above the receptacle, a cooling coil located in said container, a mass of inorganic fibrous material packed around said cooling coil, means for controlling the temperature of the cooling fluid in said cooling coil by the temperature of the condenser, and connections whereby the still vapors pass through the fibrous material on their way to the condenser.

6. In a distilling apparatus, the combination, with a still, of a condensate receptacle located within the upper part of the still, a wall of heat insulating material surrounding the receptacle, a condensing worm located within the receptacle and having its lower end arranged to discharge into the receptacle, a cooling coil telescopically arranged in said condensing worm, and having an outlet at its upper end, a second cooling coil located in the receptacle and having an inlet at its upper end, the lower ends of the cooling coils being connected, and means for conducting vapors from the still to the upper end of the condensing worm.

7. In a distilling apparatus, the combination, with a still, of a condensate receptacle located within the upper part of said still, a wall of heat insulating material surrounding said receptacle, condensing means located in said receptacle, and connections between the still and said condensing means whereby the still vapors are condensed and discharged into the condensate receptacle.

8. In a distilling apparatus, the combination, with a still, a condensate receptacle located within the upper part of the still, and a wall of insulating material surrounding the receptacle, of a condensing worm located within the receptacle and having its lower end arranged to discharge into said receptacle, a cooling coil telescopically arranged in said condensing worm and having an outlet at its upper end, a second cooling coil located in said receptacle and having an inlet at its upper end, the aforesaid two cooling coils having their lower ends connected, a container located above the receptacle, a third cooling coil in said receptacle and having its inlet connected with the outlet of the telescopically arranged cooling coil, a mass of inorganic fibrous material in which said third cooling coil is embedded and means for conducting vapors from said still through the said fibrous material and to the upper end of the condensing worm.

9. In a distilling apparatus, the combination, with a still, of a condensate receptacle located within the upper part of the still, condensing means located within and arranged to discharge the condensate into said receptacle, a container located above the receptacle, a cooling coil located in said container, a mass of inorganic fibrous material packed around said cooling coil, connections between the still and the container and between the container and the condensing means whereby the still vapors pass through the fibrous material on their way to the condensing means, and a valve in the connections between the still and container.

10. In a distilling apparatus, the combination, with a still, a condenser, a mass of sub-divided packing material arranged to be traversed by the vapor on its way from the still to the condenser, and means for cooling said sub-divided packing material, of a plurality of diaphragms located in the

still and arranged to be traversed by the vapor prior to its arrival at the said subdivided packing material.

11. In a distilling apparatus, a still chamber, a chamber thereabove and in free communication therewith, means for removing vapor, means for condensing such vapor, said means including a cooling coil, a cooling coil in the upper chamber connected to the stated cooling coil to receive cooling fluid therefrom and a packing of fibrous mineral matter in the upper chamber and in contact with the second cooling coil.

In witness whereof I have hereunto affixed my hand this 15th day of September 1911.

CLYDE E. MOLESWORTH.

Witnesses:
M. C. MASSIE,
JOHN H. SIGGERS.