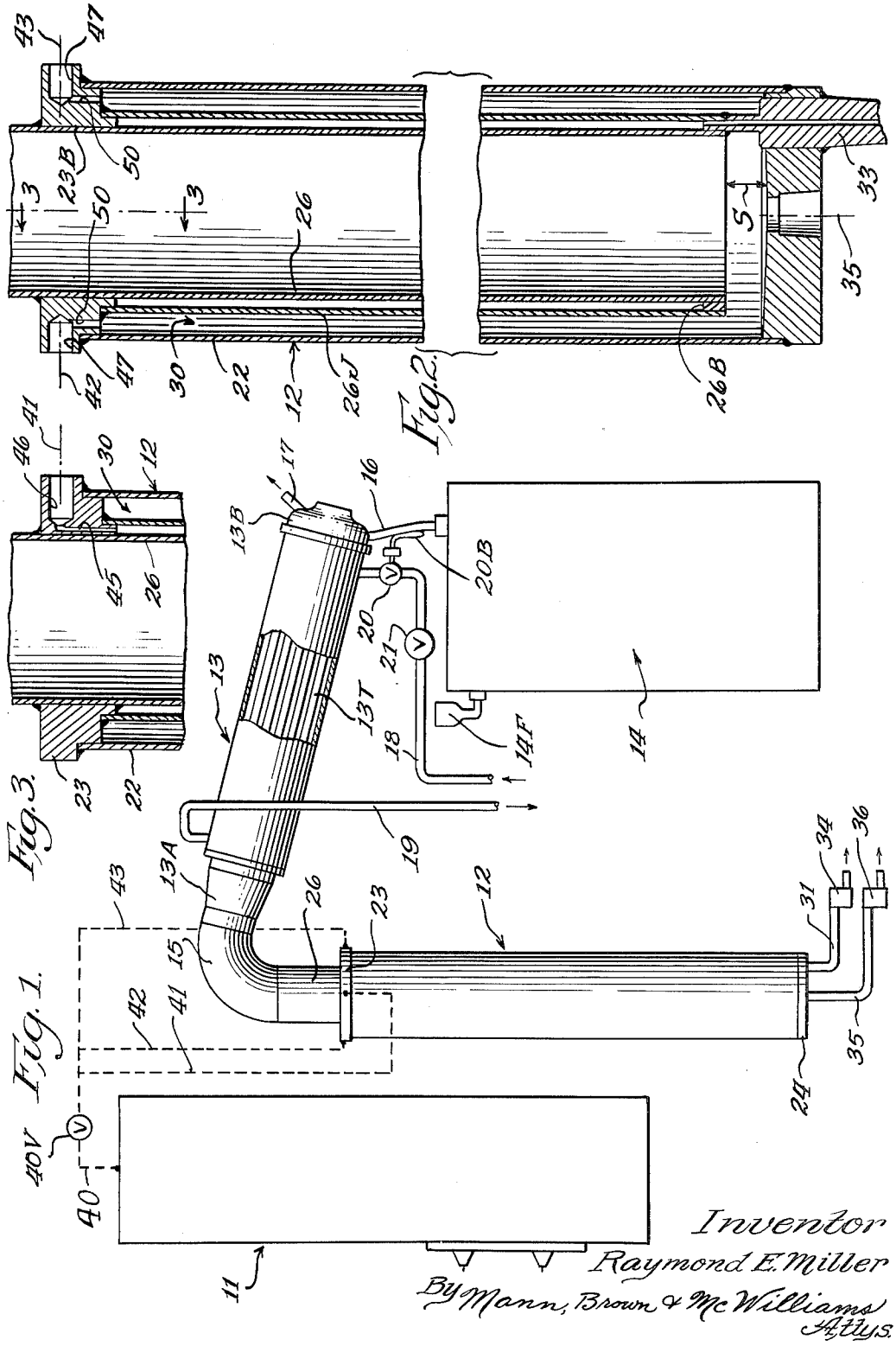
Feb. 15, 1966 R. E. MILLER 3,235,470

METHOD AND APPARATUS FOR PRODUCING HIGH PURITY DISTILLED WATER

Filed April 29, 1964



United States Patent Office 3,235,470 Patented Feb. 15, 1966

3,235,470

METHOD AND APPARATUS FOR PRODUCING HIGH PURITY DISTILLED WATER

Raymond E. Miller, R.R. 3, P.O. S10W27275, Waukesha, Wis.

Filed Apr. 29, 1964, Ser. No. 363,542
17 Claims. (Cl. 203—11)

This application is a continuation-in-part of my co-pending application Serial No. 90,258, filed February 20, 1961, and the disclosure thereof, to the extent that is not inconsistent herewith, is hereby herein incorporated by reference.

This invention relates to the production of distilled water, and particularly to the production of sterile, pyrogen-free distilled water of high chemical purity.

Distilled water of the aforesaid character is essential for many hospital and medical supply activities, and while stills for the productions of distilled water requiring such purity are commercially available and are widely used, it is nevertheless true that in such prior stills, the attainment of the desired standards is dependent upon mechanical baffles, vapor scrubbers and the like which must be replaced, or cleaned and sterilized at frequent intervals to maintain the required output standards. Moreover, such prior stills employ evaporators, operating at atmospheric pressure, and with such apparatus there is severe and rapid scale production in the evaporator, as well as in the condenser, and as a result frequent cleaning of the apparatus is essential.

In view of the foregoing it is the primary object of this invention to simplify the production of sterile, pyrogen-free distilled water, and an object related to the foregoing is to enable such results to be attained without the use of mechanical baffles or vapor scrubbers.

Another important object is to enable high quality distilled water of the aforesaid character to be produced in such a way that sterilization of the distilling apparatus may be readily and easily accomplished without disassembly and merely as an incident to the start-up operation of the apparatus. A further and related object of this invention is to produce such high quality distilled water by a new and improved method which eliminates the necessity for disassembly of the apparatus for cleaning.

Other and further objects of the present invention will be apparent from the following description and claims, and are illustrated in the accompanying drawings, which, by way of illustration, show preferred embodiments of the present invention and the principles thereof, and what is now considered to be the best mode in which to apply these principles. Other embodiments of the invention embodying the same or equivalent principles may be used and structural changes may be made as desired by those skilled in the art without departing from the invention.

In the drawings:

FIG. 1 is an elevational view of distilling apparatus embodying the features of the invention;

FIG. 2 is an enlarged fragmental vertical sectional view of the decontamination tower; and

FIG. 3 is a fragmentary cross sectional view taken along the line 3—3 of FIG. 2.

For the purposes of disclosure the invention is herein illustrated as embodied in distilling apparatus for producing sterile pyrogen-free distilled water, and this apparatus, in accordance with the present invention, derives a constant supply of saturated steam at a constant sterilizing temperature from a steam source such as a boiler **11**, and this steam, under controlled conditions as will be described, passes through a decontamination tower **12** and thence through a vented, water cooled condenser **13** from which the condensed output is discharged into a storage

container **14** having the usual protective vent and filter means **14F** associated therewith to protect the stored distillate against airborne contamination.

The condenser **13** is of the shell and tube type having one end housing **13A** forming the vapor inlet and connected through an elbow **15** to the upper end of the outlet of the decontamination tower **12**, as will be described. The water vapor entering the end housing **13A** passes through the tubes **13T** of the condenser **13**, and condensate collected in the other end housing **13B** is drained through a discharge line **16** into the distillate storage container **14**. The end housing **13B** has an upper vent **17** through which uncondensed vapor is dissipated. A cooling circuit in counter-flow relation is provided by means including inlet and outlet lines **18** and **19** connected to opposite ends of the condenser shell, and to control the system operation, an automatic valve **20** in the water inlet line **18** is thermostatically controlled by means including a temperature sensing bulb **20B** that senses the output temperature of the distillate in the output line **16**. The thermostatic control is set to such a value that there is at all times a slight amount of uncondensed vapor being vented at **17**. A second valve **21** in the water inlet line **18** is used during start-up operation to temporarily prevent cooling of the condenser as will be described.

The steam source such as the boiler **11** must of course provide a constant steam supply at the desired pressure, and since there is no condensate return, unusually heavy treatment of the make-up water is required in order to avoid the necessity of frequent blow-down operation to maintain satisfactory boiler operation. A highly satisfactory boiler treatment additive is provided by a formulation having sodium polyphosphate 60%, monosodium phosphate 20%, sodium acid pyrophosphate 10% and sodium sulfite 10%. This serves as a non-toxic combination of sequestering and complexing agents, phosphatizing agents, and also acts as a reducing agent to remove dissolved oxygen from the water.

This additive is preferably supplied to the boiler make-up water at about 100 parts per million, but this proportion may be varied, in accordance with the make-up water used, to the level required to prevent liming or scale formation in the boiler. The same additive is preferably employed at a rate of about 10 parts per million in the cooling water so as to prevent liming of the related surfaces of the condenser **13**.

The decontamination tower **12** comprises an outer cylindrical wall **22**, FIGS. 2 and 3, closed at its top and bottom ends by top and bottom plates **23** and **24** that are welded to the ends of the outer wall. The top plate **23** has a central opening **23B** through which a central tube **26** extends in a sealed and welded relation, and the upper end of the tube **26** is connected by welding to the lower end of the vapor inlet elbow **15**. The central tube **26** extends downwardly in coaxial relation to the outer wall **22** to a level relatively close to but spaced from the bottom wall **24** in a dimension S. The tube **26** provides a steam delivery conduit discharging upwardly into the vapor supply pipe **15**, and means are provided for constantly heating the conduit to a sterilizing temperature. Such heating means in the present instance comprises a steam jacket provided by a tubular member **26J** surrounding the tube **26** in spaced relation and secured as by welding at its upper to the top plate **23** concentric with the tube **26** and connected at its lower end to the tube **26** by a cross wall **26B** that is welded in position.

The tubular member **26J** is also spaced from the outer wall of the tower **12** so as to provide an annular outer passage **30** communicating with the lower end of the steam delivery conduit **26** through the annular area de-

fined between the bottom wall **24** and the cross wall **26B** of the steam jacket. The steam from the boiler **11** is introduced at the upper end of the outer passage **30**, as will be described, and flows downwardly in the passage **30**, through the annular area above mentioned, and then upwardly through the conduit **26**, and the vapor inlet elbow **15** to the condenser **13**, and best results are attained by proportioning these parts so that the steam passage provided thereby is of substantially uniform area throughout. This causes the steam to travel at a uniform speed throughout the length of the passage.

The steam jacket has a drain line **31** connected thereto by a fitting **33** extended through the bottom wall **26B** and the bottom plate **24** in welded relation, and the drain line **31** is extended from the fitting **33** through a steam trap **34** for draining condensate from the steam jacket. Similarly, the bottom plate **24** has its upper surface sloped to the center of the plate and a drain line **35** through the center of the plate discharges condensate through a steam trap **36**.

Saturated steam at the desired sterilizing temperature (preferably 24–25 pounds per square inch pressure) is supplied by the boiler **11** through a steam line **40**, and where the heating means for the steam conduit **26** is in the form of a steam jacket, a branch steam line **41** from the line **40** is arranged to discharge steam at substantially its original temperature into the upper end of the steam jacket, while a pair of branch lines **42** and **43** are connected through throttling means to discharge steam at substantially atmospheric pressure and in a superheated state into the upper end of the annular outer passage **30** of the decontaminating tower. In accomplishing this the top plate **23** of the tower is utilized to provide the terminal portions of the steam lines **41**, **42** and **43**.

Thus, as shown in FIG. 3, a blind vertical bore **45** extended upwardly into the top plate **23** opens at its lower end into the space between the tubes **26** and **26J**, and intersects at its upper end with a blind radial bore **46** to which the branch steam line **41** is connected.

At opposite sides, as shown in FIG. 2, the top plate **23** has blind radial bores **47** extended into the edges of the plate and these bores are connected to the respective steam lines **42** and **43**. Steam supplied to the bores **47** at substantially its original pressure is throttled into opposite sides of the passage **30** by restricted trottling orifices **50** formed as blind upward bores in the plate **23** and intersecting the respective radial bores **47**. The orifice size is chosen with relation to the original steam pressure so as to throttle the steam into the passage **30** at substantially atmospheric pressure.

In the operation of the present distilling apparatus, the steam is supplied to the line **40** from the boiler **11** as saturated steam having a temperature of from 260° F. to 265° F. which is usually considered to be a temperature at which sterilization takes place instantaneously. This saturated steam flows through the branch lines **42** and **43** with some loss of heat but reaches the throttling orifices **50** at a temperature of substantially 250°. This saturated steam is throttled into the upper end of the annular passage **30** on opposite sides thereof so that upon entry into the passage **30**, this steam is at substantially atmosphere pressure and still maintains substantially 250° F. as its temperature and of course possesses considerable superheat. The superheated steam travels downwardly at high speed within the annular passage **30** as will be described in some detail hereinafter, and in such travel is supplied with heat from the steam jacket wall **26J** that defines one boundary of the annular passage **30**. The steam jacket of course is heated by the continuous steam supply from the branch line **41**, and as the steam within the steam jacket condenses, the condensate is withdrawn through the steam-trapped waste line **31**.

By reason of the throttling of the steam into the inlet end of the annular passage **30**, any unvaporized moisture carried by the steam is immediately vaporized, and as the steam travels downwardly through the annular passage **30**, the constant supply of heat from the steam jacket minimizes the net heat loss from this steam so that when the steam reaches the lower end of the annular passage **30** its temperature remains well above 212° F. and, in fact, is relatively close to or slightly above a sterilizing temperature.

Such steam passes through the space between the lower end of the steam jacket and the bottom plate of the tower and then passes upwardly through the heated steam conduit **26**. During this upward passage, heat is also supplied to the moving steam and it is found that as the steam moves out of the tower **12** and into the vapor inlet of the condenser **13**, its temperature is still in the neighborhood of a sterilizing temperature of 230° F.

The steam then passes through the tubes **13T** of the condenser and condensate is withdrawn through the condensate line **16**. Any uncondensed steam of course is released to atmosphere through the vent **17** in the end housing **13B** of the condenser.

In the start-up operation of the present apparatus, a steam valve **40V** in the line **40** is opened and the manual water shut off valve **21** in the cooling circuit is closed. Steam is thus supplied at well above a sterilizing temperature to the entire system, such steam flowing through the annular passage **30** and being supplied with additional heat from the steam jacket. The steam flow continues through the conduit **26**, and through the tubes **13T** of the condenser, this steam being discharged through the vent **17** and through the condensate line **16** which under such circumstances is disconnected from the container **14**. This flow of sterilizing steam is allowed to continue through the system for a sterilizing cycle of from 5 to 30 minutes, and of course it is recognized that initially the entire apparatus must be heated to what may be termed its normal operating temperature. During the later portions of such start-up operation, the various internal parts of the system of course reach a temperature relatively close to the temperature of the steam, and these internal surfaces are thus sterilized during the start-up operation. In this respect the fact that the steam initially supplied is at the temperature well above the minimum sterilizing temperature constitutes an important consideration.

After the apparatus has thus been sterilized and brought up to proper working temperatures throughout, the manual valve **21** in the cooling circuit is opened and after the condenser has been cooled to its working temperature, the production of high quality distilled water begins. At this time, the distillate line **16** is connected to the distillate container **14**.

It has been found that where the present apparatus is operated in the manner above described, the resulting distillate is sterilized and is pyrogen-free. Further it is found that the resulting distillate has an electrical resistance far above the minimum requirements usually imposed, and the dissolved solids are well below the maximum limits imposed for the most exacting uses.

In operation of distilling apparatus constructed as above described, and having an output capacity of ten gallons per hour, it has been determined that substantially continuous operation for about one year produced no appreciable deposits of any kind on the internal surfaces of the decontamination tower or on the condensing surfaces of condenser, and present indications are that the tower and the condenser as employed under this invention may be used almost indefinitely without the necessity for internal cleaning.

From the foregoing description it will be apparent that the present invention provides a new and improved method of producing high purity distilled water, and that under this method the production of distilled water is materially simplified because the apparatus may operate

for extremely long periods without the necessity for cleaning the apparatus.

It will also be apparent that the present invention invention enables extremely high quality distilled water to be produced without the necessity for using mechanical baffles or vapor scrubbers.

Thus while a preferred embodiment of the invention has been illustrated herein, it is to be understood that changes and variations may be made by those skilled in the art without departing from the spirit and scope of the appended claims.

I claim:

1. In a distilling system for producing, sterile, pyrogen-free distilled water, a condenser having a thermostatically controlled cooling circuit and a condensing circuit including a steam input end, a vented condensate output end and a condensate delivery line at said output end, a vertically elongated decontamination tower having a main housing with a cylindrical outer wall closed at its ends by top and bottom walls and a steam delivery conduit extending through the top wall and discharging at its upper end into said input end of the condenser, said steam delivery conduit being extended downwardly within said outer wall in centered and spaced relation to a level spaced a short distance from the bottom wall to define, with said cylindrical wall, an annular outer passage surrounding the conduit and communicating with the steam delivery conduit through the space between the bottom wall and the lower end of the conduit, means associated with said conduit within the housing for heating said conduit to a sterlizing temperature, means for throttling saturated steam from an elevated pressure that provides a sterilizing temperature into the upper portion of the outer passage at substantially atmosphere pressure, a steam trapped drain opening through the bottom wall of the housing for withdrawing condensate formed uring start-up operation.

2. In a distillation apparatus for producing sterile, pyrogen-free distilled water, a condenser having a vapor inlet, a vapor outlet and a condensate outlet and having a coolant circuit, a decontamination tower comprising a vertically elongated housing with upper and lower closure plates to define a main steam chamber in the housing, an elongated tubular member open at both ends disposed vertically in said steam chamber and extended through said upper closure plate, a steam conduit extended upwardly from the open upper end of said tubular member and connected to the vapor inlet of said condenser and through which steam passes from the housing to the vapor inlet of the condenser, said tubular member having its lower end spaced from the lower closure plate of the housing so that steam from said main steam chamber may enter the lower end of the tubular member and pass upwardly through the tubular member from said main steam chamber and into said steam conduit, steam jacket means associated with said tubular member for heating the same, means for supplying superheated steam at a selected pressure to the upper portion of the main steam chamber, and means for supplying steam to said steam jacket at a pressure higher than said selected pressure whereby to supply heat to the steam moving through said steam chamber.

3. In a distillation apparatus for producing sterile, pyrogen-free distilled water, a condenser having a vapor inlet, a vapor outlet and a condensate outlet and having a coolant circuit, a decontamination tower comprising a vertically elongated housing with upper and lower closure plates to define a main steam chamber in the housing, an elongated tubular member open at both ends disposed vertically in said steam chamber and extended through said upper closure plate, a steam conduit extended upwardly from the open upper end of said tubular member and connected to the vapor inlet of said condenser and through which steam passes from the housing to the vapor inlet of the condenser, said tubular member having its lower end spaced from the lower closure plate of the housing so that steam from said main steam chamber may enter the lower end of the tubular member and pass upwardly through the tubular member from said main steam chamber and into said steam conduit, means associated with said tubular member for heating the same to a sterilizing temperature, and means for supplying superheated steam to the upper portion of the main steam chamber.

4. In distillation apparatus for producing sterile, pyrogen-free, distilled water, a vertically elongated decontamination tower having a cylindrical outer wall, top and bottom closure walls, steam conduit means within said decontamination tower including a tubular member spaced from said outer wall to define an annular space therebetween and spaced from said bottom wall to define a continuous steam passage from the outer annular space to the space within said tubular member, said tubular member extending upwardly through the top wall, means for heating said tubular member to a sterilizing temperature, means for throttling saturated steam from an elevated pressure into said annular space, means for condensing steam issuing from the upper end of said tubular member and collecting said condensate and a drain opening through the bottom wall of the tower for withdrawing condensate from the tower.

5. Distillation apparatus according to claim 4 in which said heating means consists of a steam jacket which constitutes the wall of said tubular member.

6. In distillation apparatus for producing sterile, pyrogen-free, distilled water, a vertically elongated decontamination tower comprising a cylindrical outer wall, a concentric tubular member of smaller diameter than the outer wall mounted within the cylindrical wall and defining an annular space therebetween, top and bottom closure walls for the tower with the top wall having an opening therethrough in communication with the space within said tubular member, said tubular member being spaced from the bottom wall to provide communication between said annular space and the space within the tubular member, means for heating said conduit to a sterilizing temperature, means for throttling saturated steam from an elevated pressure to a substantially lower pressure into the upper portion of said annular space, means for condensing steam isuing from the upper end of said tubular member and collecting said condensate and a steam-trapped drain opening through the bottom wall of the tower for withdrawing condensate from the tower.

7. In apparatus for producing sterile, pyrogen-free, distilled water, the combination of boiler means for heating water under pressure to a temperature of at least 250° F. to produce saturated steam, a decontamination tower comprising an outer cylindrical wall and a tubular member of lesser diameter mounted concentrically within said cylindrical wall, top and bottom closure walls for the tower, the top wall having an opening therethrough in communication with the interior of said tubular member, said tubular member being spaced from said bottom wall to provide communication between the interior of the tubular member and the annular space between the tubular member and said cylindrical wall, means for introducing steam from said boiler into the upper part of said annular space through a throttling orifice to cause complete vaporization of said steam, means for heating said tubular member to a temperature above sterilizing temperature and maintaining it at said temperature during operation of the tower, and means for condensing steam issuing from the upper end of said tubular passage and collecting said condensate.

8. Distillation apparatus as set forth in claim 7 in which the heating means for said tubular member maintains the latter at a sufficiently elevated temperature that the steam passing through the annular space and thence through the space within said tubular member leaves the top of

the decontamination tower at a sterilizing temperature and in superheated condition.

9. Distillation apparatus as set forth in claim **7** in which said boiler means includes a source of makeup water which has been treated with a non-toxic boiler additive.

10. Distillation apparatus as set forth in claim **9** in which the non-toxic boiler additive includes sequestering and complexing agents, phosphatizing agents and a reducing agent.

11. In distillation apparatus for producing sterile, pyrogen-free, distilled water, a vertically elongated decontamination tower having a cylindrical outer wall, top and bottom closure walls, steam conduit means within said decontamination tower including a tubular member spaced from said outer wall to define an annular space therebetween and spaced from said bottom wall to define a continuous steam passage from the outer annular space to the space within said tubular member, said tubular member extending upwardly through the top wall, means for heating said tubular member to a sterilizing temperature, means for throttling saturated steam from an elevated pressure into said annular space, and a drain opening through the bottom wall of the tower for withdrawing condensate, said throttling means including discharge orifices in said top wall at spaced points about said annular space.

12. Distillation apparatus as set forth in claim **11** in which said outer annular space has substantially the same cross-sectional area as the space within said tubular member, whereby said continuous steam passage causes a substantially constant flow rate of the steam as it passes through the apparatus.

13. The method of producing sterile, pyrogen-free, distilled water which consists in heating a body of water under super-atmospheric pressure to vaporize a portion thereof into saturated steam at a sterilizing temperature and under super-atmospheric pressure, taking said saturated steam and dividing it into two lines reducing the pressure in one of said lines to substantially atmospheric pressure while it is passed through a conduit that is maintained above said sterilizing temperature by steam from said second line, condensing the steam in said first line, and collecting the condensate.

14. The method of treating steam for the production of sterile, pyrogen-free, distilled water which consists in taking saturated steam at a temperature of at least 250° F., and at super-atmospheric pressure, expanding it to substantially atmospheric pressure through a conduit which is continuously maintained by external means at a temperature above sterilizing temperature to thereby produce superheated steam at sterilizing temperature, and then condensing said steam.

15. The method as set forth in claim **14** in which the steam when expanded to substantially atmospheric pressure is flowed in one direction, thence through a reverse bend, and thence in an opposite direction along an elongated confined path toward a vented condenser at a substantially uniform velocity.

16. The method of treating steam for the production of sterile, pyrogen-free, distilled water which consists in taking steam at a temperature of at least 250° F., and at super-atmospheric pressure, flash vaporizing the steam into a conduit which is selectively heated by external means to such a temperature in relation to said vaporized steam as to prevent the steam temperature in the conduit from dropping below 212° F. while passing through a first portion of the conduit, then reheating the steam as it passes through a second portion of the conduit to a temperature which gives the steam substantial superheat, collecting and removing the condensate formed within the conduit, and then condensing the reheated superheated steam and collecting the distillate therefrom.

17. The method as set forth in claim **14** in which the saturated steam is produced from water that has been treated with a non-toxic boiler additive.

References Cited by the Examiner

UNITED STATES PATENTS

1,280,851	10/1918	Rogers	165—155
2,372,079	3/1945	Gunter	165—155
3,087,253	4/1963	Wulf	165—155

NORMAN YUDKOFF, *Primary Examiner.*