

Feb. 20, 1945.

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2,369,900

METHOD AND APPARATUS FOR STERILIZING AIR

Filed March 31, 1944

3 Sheets-Sheet 1

Fig. 1.

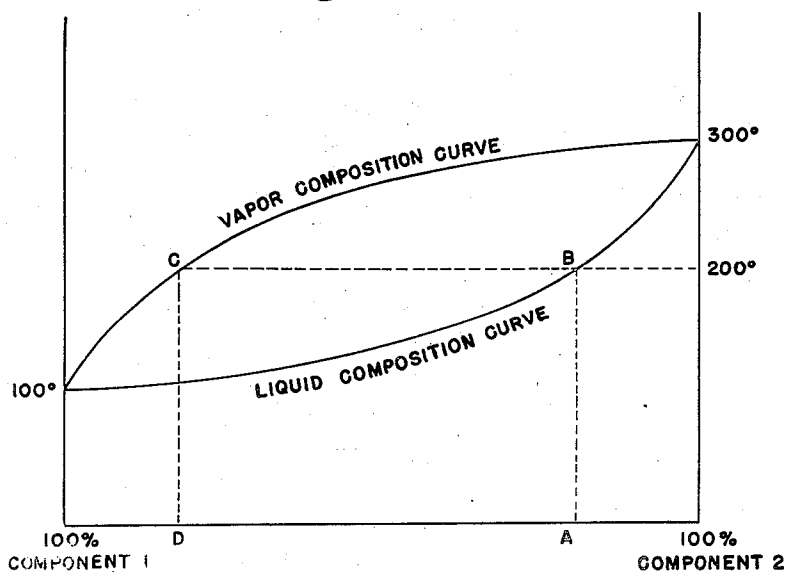
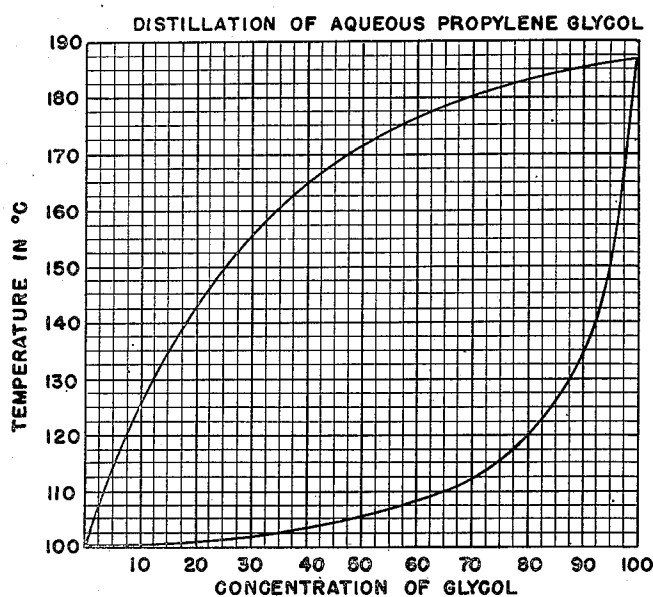


Fig. 2.



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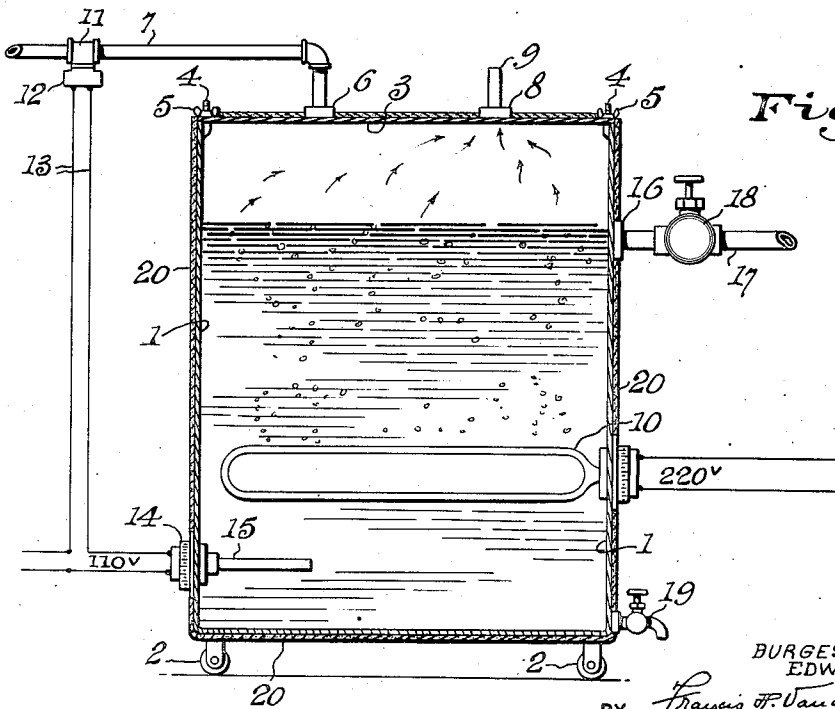
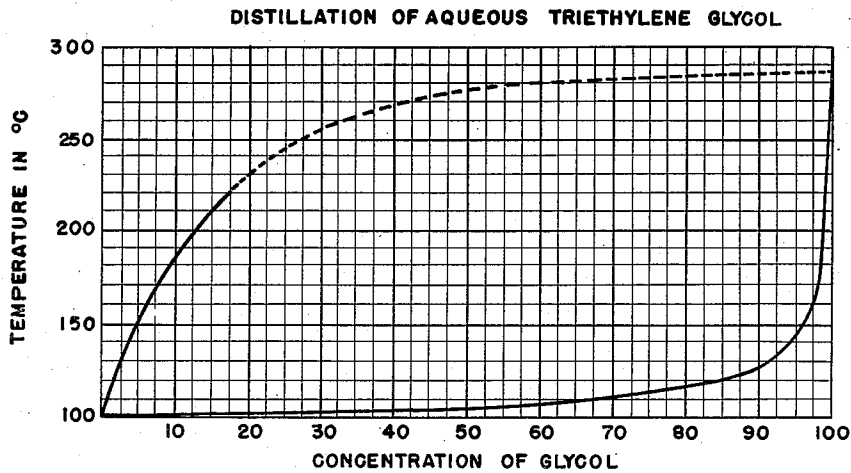
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Fig. 3.



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Fig. 6.

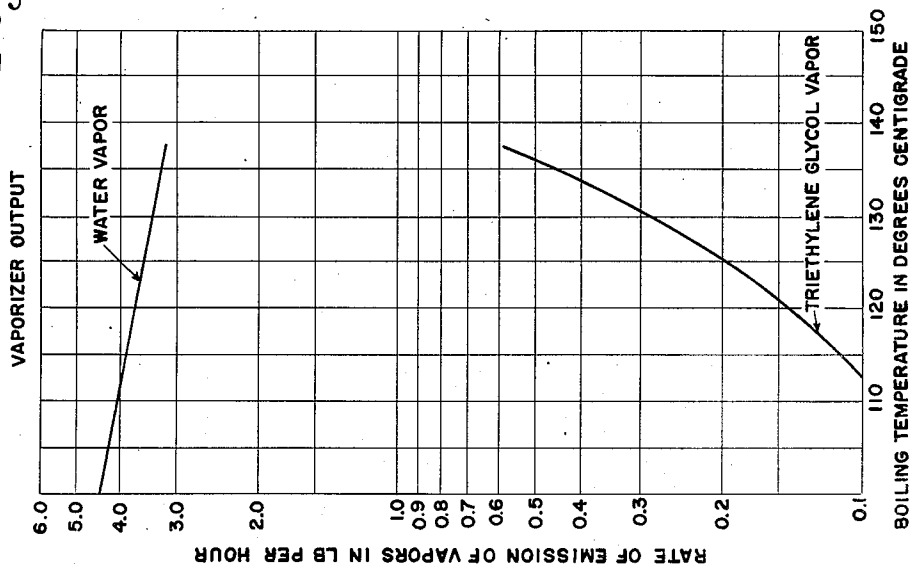
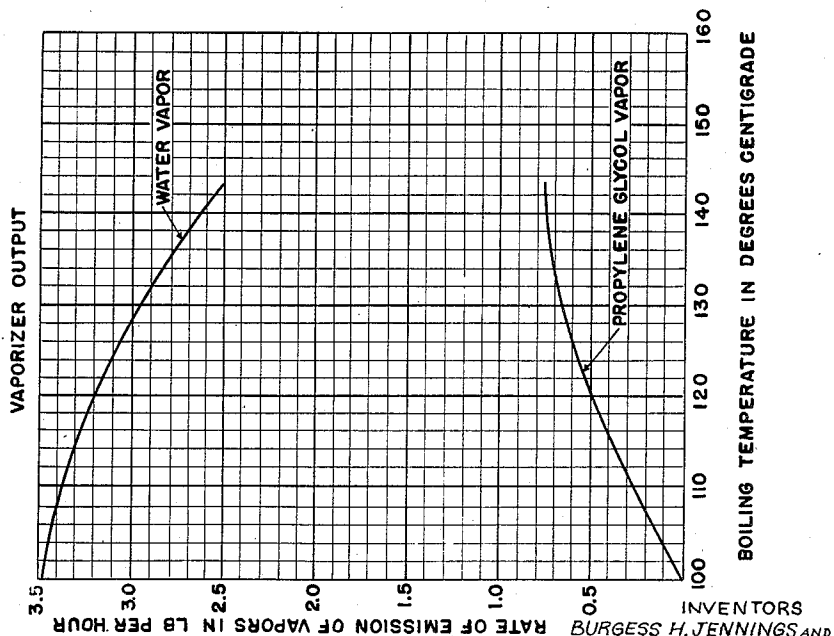


Fig. 5.



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METHOD AND APPARATUS FOR
STERILIZING AIR

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Application March 31, 1944, Serial No. 528,988

19 Claims. (Cl. 21—53)

This invention relates to methods and apparatus for sterilizing air by means of vapors and more particularly to a method and apparatus for introducing measured quantities of water vapor and vapor of non-germicidal materials into the atmosphere.

Numerous methods have been utilized to sterilize air by means of germicidal liquid aerosols, but among these methods none has been entirely satisfactory. The most effective of these prior attempts consisted of introducing a solution of a germicidal agent such as, a solution of resorcinol or hexyl resorcinol in a suitable solvent in droplet form into the air. These agents, as well as other germicidal agents, were found to possess an objectionable degree of toxicity. This important objection to such methods was overcome by the method disclosed in Patent Number 2,333,124, issued November 2, 1943, to Oswald H. Robertson, Benjamin F. Miller and Edward Bigg, which comprises bringing air laden with bacteria or other pathogenic organisms into contact with a non-germicidal material such as a glycol in vapor form. The patentees discovered that small amounts of glycol in vapor form in the atmosphere have a 100% lethal action on the bacteria in the air without producing any local or systemic effects upon the occupants of an enclosure, even when inhaled continuously for prolonged periods of time. While this method represents an outstanding improvement over the methods of the prior art, it has been determined that heated glycol vapors create fire hazards owing to their flammable nature and explosive characteristics. Furthermore, it has been ascertained that the lethal action of glycol vapors on bacteria-laden air varies with the relative humidity of the air and under controlled humidity conditions exhibit even greater lethal action on bacteria than when used under the low humidity conditions which usually prevail in living quarters.

This invention is an improvement over the invention disclosed in the above patent and comprises a method and means of introducing measured quantities of glycol and water vapors into the atmosphere at predetermined rates. Glycol vapor exerts its greatest bactericidal activity on air-borne organisms in the presence of a relative humidity of from 35 to 45 per cent. In most living quarters equipped with conventional heating systems, low humidities prevail with the result that cross infections occur readily. The present invention provides a method of introducing measured quantities of glycol and water

vapor into the air at predetermined rates to overcome such conditions and also provides for an apparatus for accomplishing the foregoing, thus eliminating the necessity for using a separate humidification system to enhance the bactericidal activity of the glycol vapor.

This invention has for its object a method of sterilizing air by introducing measured quantities of glycol and water vapors into the atmosphere at predetermined rates.

Another object of this invention is to provide an apparatus for simultaneously introducing measured quantities of glycol and water vapors into the atmosphere at predetermined rates.

Still another object of this invention is to eliminate fire hazards in rooms or other enclosures arising from the introduction of heated glycol vapor into the atmosphere in quantities sufficient to exhibit a lethal action upon bacteria and other pathogenic organisms in the air.

A further object of this invention is to provide a method and apparatus for sterilizing air by heating aqueous glycol solutions at predetermined constant temperatures and for controlling the concentrations of the components of both the liquid and vapor phases of the system as well as the rate of input of heat to the liquid phase.

A still further object of this invention is to provide a simple and inexpensive apparatus for sterilizing air containing bacteria.

Other objects will be apparent from the following description of the invention and the accompanying drawings in which:

Figure 1 is a distillation diagram for a theoretical ideal binary solution showing the relation of boiling point and composition between the liquid and vapor phases;

Figure 2 is a distillation diagram for an aqueous propylene glycol system showing the relation of boiling point and composition between the liquid and vapor phases;

Figure 3 is a distillation diagram for an aqueous triethylene glycol system showing the relation of boiling point and composition between the liquid and vapor phases;

Figure 4 is a side elevational view in cross section of the sterilizing apparatus;

Figure 5 is a diagram showing the total output of propylene glycol vapor and water vapor by the sterilizing apparatus at various temperatures of the liquid phase; and

Figure 6 is a diagram showing the total output of triethylene glycol vapor and water vapor by the sterilizing apparatus at various temperatures of the liquid phase.

In practicing this invention an aqueous glycol solution is boiled to produce glycol and water vapor which is brought into contact with air containing bacteria and other pathogenic organisms. The proportion of glycol and water vaporized depends upon their respective concentrations in the liquid phase and the rate of introduction into the atmosphere is determined by the rate of heat input or the rate of boiling. Since the action of boiling any aqueous solution for a time produces a change in the composition of the liquid and vapor phases of the system, it is contemplated by this invention to maintain the composition of the liquid phase constant and, by so doing, regulate the proportions of glycol and water in the vapor phase. Considering the theoretical basis of the foregoing, when two or more perfectly miscible liquids are dissolved in each other to form an ideal solution, the vapor pressure of any component and its proportion in the vapor phase has a definite relationship to the mole fraction of that component in the liquid phase. This relationship is known as Raoult's law and for an ideal binary solution is stated as follows:

(I)

$$P_1 = P_1^0 \frac{n_1}{n_1 + n_2} = P_1^0 N_1 \text{ and } P_2 = P_2^0 \frac{n_2}{n_1 + n_2} = P_2^0 N_2$$

where P_1 and P_2 are the pressures of components 1 and 2, in the vapor phase at a given temperature; P_1^0 and P_2^0 are the vapor pressures of components 1 and 2, in the pure state at the given temperature; N_1 and N_2 are the mole fractions of components 1 and 2; and n_1 and n_2 are the number of moles of components 1 and 2, present in the solution. The total vapor pressure above a solution is the sum of the partial pressures of the components and depends upon the particular liquids, their relative proportions and temperature. The boiling point of a mixture at a given pressure is the temperature at which the sum of the partial pressure of the components equals that pressure. Thus, at atmospheric pressure, $P_1 + P_2 = 760$ mm. Hg or 14.7 pounds per square inch. With rising temperature, the vapor pressure of a solution increases and the partial pressure of each component of a binary system will be

(II)

$$P_1 = P_1^t \frac{n_1}{n_1 + n_2} = P_1^t N_1 \text{ and } P_2 = P_2^t \frac{n_2}{n_1 + n_2} = P_2^t N_2$$

where P_1^t and P_2^t are the vapor pressures of the components 1 and 2 in the pure state at the temperature t .

It is apparent from the above discussion that a solution containing a larger proportion of a volatile component will have a lower boiling point than a solution containing a smaller amount. Referring to Figure 1, which shows the distillation diagram of an ideal binary solution consisting of components 1 and 2, it will be seen that a composition of the mixture at a point such as A will have a boiling point corresponding to B and conversely a mixture of components 1 and 2 that boils at temperature B corresponds to the composition at point A. The composition of the vapors will be different than the composition of the solution with which they are in equilibrium and, as indicated by the vapor composition curve in Figure 1, the vapors will be richer in the more volatile constituent or component 1. This follows from the action of boiling a mixture corresponding to A for a time and leading off the vapors as in a distillation process. The liquid remaining behind in the boiler will become richer

in the less volatile constituent and, as the removal of the more volatile constituent continues, the boiling point of the solution rises until all of the more volatile constituent has been removed.

When this point is reached the temperature becomes stationary at the boiling point of the less volatile constituent. The composition of vapors in equilibrium with a particular solution such as the mixture corresponding to A in Figure 1 may be determined by projecting a vertical line from the point A to the liquid composition curve at the point B. A horizontal line is then drawn until it intersects the vapor composition curve as at the point C. A vertical line is dropped from the point C to the point D which gives the composition of the vapor in equilibrium with the solution at point A.

The method contemplated by this invention is essentially a distillation and it is necessary to determine the liquid and vapor composition curves for the glycol-water systems used, to control the quantities of glycol and water vapor introduced into the atmosphere. While these diagrams may be developed by calculations employing Equation II above, it is more accurate to derive the data experimentally, since the theoretical generalizations apply only to ideal solutions and most solutions deviate substantially therefrom. While any of the glycols may be used, it is preferred to employ propylene glycol and triethylene glycol. Any standard distillation apparatus may be employed to obtain the temperature-composition data of the liquid and vapor phases of propylene glycol-water and triethylene glycol water systems. One standard distillation apparatus consists of a side-armed flask provided with a pair of thermometers for indicating the temperatures of the liquid and vapor phases and a Liebig condenser which may be connected to the side-arm of the flask in either refluxing or distilling positions. In using this apparatus the flask was filled with the aqueous glycol solution and the solution refluxed until the liquid and vapor temperatures remained constant. The condenser was then moved to the distilling position and a 4 or 5 cubic centimeter sample distilled from the flask. The heat was removed and a sample of the liquid in the flask obtained when the boiling had ceased. The temperature at which the samples were taken is noted and the process repeated until only pure glycol was obtained in the distillate. The corresponding set of flask and distillate samples were analyzed for glycol and water content. The samples from the flask were analyzed by comparative viscosity tests against standardized samples, while the distillates were analyzed by refractive index determinations. These methods are rapid, convenient and accurate to within 0.1%. From the temperatures and the analysis of the corresponding flask and distillate samples it was possible to construct the temperature-composition distillation curves for propylene glycol-water and triethylene glycol-water systems shown in Figures 2 and 3, respectively. The data used in preparing these curves, while approximate, were obtained under conditions closely simulating the actual operation of the sterilizing apparatus contemplated by this invention.

Referring to Figure 2 which shows the distillation curve for propylene glycol-water systems, it is apparent that glycol vapor concentrations from 0-100% may be obtained from aqueous propylene glycol solutions. It has been determined that in order to introduce the quantity of propylene gly-

col vapor into the atmosphere necessary to assure bactericidal effects and the quantity of water necessary to provide a relative humidity at which the bactericidal properties of the glycol vapor are most effective, the aqueous propylene glycol solution must be maintained at a maximum temperature of 150° C. From Figure 2 it is apparent that at temperatures of from 140°–150° C. a 95% solution boils, giving a vapor containing 25% propylene glycol and 75% water. In Figure 3 the distillation data for triethylene glycol-water systems were carried only to a point where it was evident that concentrations of from 0–18% triethylene glycol vapor could be introduced into the atmosphere. For sterilization purposes a maximum temperature of 140° C. of an aqueous triethylene glycol solution is adequate. This corresponds to the boiling point of a 93.5% solution giving a vapor containing 3% triethylene glycol and 97% water. The above operating temperatures are only maximum temperatures and lower temperatures may be utilized without decreasing the bactericidal effect of the glycol vapor. At lower temperatures a larger water vapor output is obtained which may be necessary depending upon humidity conditions of the atmosphere under treatment. The temperature and relative humidity of the atmosphere in the enclosure to be treated may be ascertained and the quantity of water vapor required to raise the relative humidity to the desired level of from 35%–45% then calculated. With knowledge of the quantity of water vapor required, an aqueous solution of glycol may be selected from the distillation curve which is in equilibrium with vapors of glycol and water, the composition of which is such that the water vapor requirement is satisfied. An aqueous glycol solution having the predetermined concentration is prepared and heated at its boiling point temperature. The vapors formed are introduced into the atmosphere and, as the temperature of the solution rises due to loss of water, additional water is added to the solution to maintain the concentration thereof constant. The glycol content of the solution is also replenished by additions thereof to the solution when necessary.

Referring to Figure 4, the preferred form of sterilizing apparatus comprises a tank 1 which contains an aqueous solution of a glycol. The tank 1 is mounted on the casters 2, to adapt it for movement and is provided with a top 3. The top 3 is provided with openings through which pass the bolts 4 formed on the tank 1. The wing nuts 5 are adapted to engage the bolts 4 and secure the top 3 on the tank 1. The top 3 is provided with a threaded opening 6 which is adapted to engage the threaded end of a water inlet pipe 7, and a second threaded opening 8, which is adapted to engage the threaded end of a vent pipe 9. An electric immersion heater 10 is mounted in the tank 1 on the wall thereof adjacent the bottom of the tank. The water inlet pipe 7 contains a flow control valve 11 which is operated by a solenoid 12. Electric current is supplied to the solenoid 12 by the circuit 13 which contains an adjustable thermo-switch 14, which is mounted on the wall of the tank 1 with the heat responsive element 15 thereof immersed in the solution in the tank. The wall of the tank 1 is provided with a threaded opening 16 which is adapted to engage the threaded end of a glycol inlet pipe 17. The pipe 17 contains a flow control valve 18. The tank 1 has a drain cock 19 at the bottom thereof to permit clean out. Heat

losses are reduced to the minimum by the insulation 20 which completely surrounds the tank 1.

In operation, the thermostatic switch is set at the temperature corresponding to the boiling point of an aqueous glycol solution of a definite concentration which is in equilibrium with vapors of water and glycol, the quantities of which humidify the atmosphere to the desired degree and exhibit a lethal action on bacteria in the atmosphere. Any concentration of aqueous glycol solution but preferably one having an excess of water is placed in the tank 1 and heated by the immersion heater 10. The solution is brought to a boil and the vapor formed flows from the tank 1 through the vent pipe 9 into the atmosphere or an air duct leading to a room or other enclosure. The glycol and water vapor continues to form and flow from the tank 1 until the concentrations of glycol and water in the liquid phase reach that of a solution having the boiling point corresponding to the temperature setting of the thermostatic switch. At this point further loss of glycol and water vapor by the liquid phase results in an increase in the concentration of the less volatile component, glycol and a corresponding increase in the boiling point temperature of the solution as indicated by the distillation curves in Figures 2 and 3. The increase in the boiling point temperature of the solution causes the thermostatic switch 14 to close the circuit 13 and actuate the solenoid 12. The solenoid 12 operates the valve 11 in the water inlet pipe 7 allowing water to flow into the tank 1. The water entering the tank 1 dilutes the solution, decreasing the concentration of the less volatile constituent and lowering the boiling point thereof. When the concentration of the aqueous glycol solution is adjusted to a point at which its boiling point temperature corresponds to the setting of the thermostatic switch 14, the latter opens and in turn operates the valve 11, stopping the flow of water through the pipe 7 into the tank 1. This process is repeated with the result that the concentration of the liquid phase is maintained at a point at which the vapors of glycol and water in equilibrium therewith contains the desired quantities of glycol and water. The cycle of operation of the sterilizing apparatus varies between 5 to 15 minutes depending upon the setting of the thermostatic switch and the rate of heat input which may be controlled by varying the voltage of the heater. This period is also reduced by the insulation on the tank 1 which minimizes heat losses. The latter influences the total output and results in a dissipation of energy which can be utilized in vaporizing the solution. The glycol content of the solution may be replaced as it is diminished by introducing additional glycol to the tank through the pipe 17. The flow of glycol is adjusted by controlling the valve 18 which may be operated manually or by a float, which is not illustrated, that is responsive to changes of the level of the liquid in the tank or by other suitable means.

The operating characteristics of the sterilizing apparatus at various temperatures using aqueous solutions of both propylene glycol and triethylene glycol are shown in Figures 5 and 6, respectively. These values are based upon total output of vapor at various temperatures over boiling periods in excess of 1000 hours. During these extended tests the concentration of glycol and water in the liquid phase and hence in the vapor phase was maintained within limits of 1% of the desired concentration.

The bactericidal activity of glycol vapor under

controlled humidity conditions was tested in a room having a volume of 10,000 cubic feet. Using filtered saliva as the diluent, 18 hour cultures of the test organism, *Staphylococcus albus* or hemolytic streptococci (*Streptococcus C*) were made up to a standard concentration of one billion organisms per cubic centimeter. Ten cubic centimeters of the same suspension were atomized under two large circulating fans for the control and test experiment. Air samples of one cubic foot were taken by means of a Moulton apparatus (S. Moulton, Science, vol. 97, page 51, Jan. 8, 1943) immediately after the bacterial spray and at 15 minute intervals for a period of two hours. The broth in the reservoir was plated and colony counts recorded. In the test, the glycol vapor was introduced from the vaporizer and air samples for the determination of numbers of bacteria and quantity of glycol were taken concomitantly. These samples were taken at various positions and levels in the room. The tests revealed that no diminution in the lethal effect of glycol vapor occurs when introduced into the atmosphere under controlled humidity conditions. Immediate death of organisms in the atmosphere was obtained by introducing 0.005 mg. per liter of triethylene glycol vapor or 0.2 mg. per liter of propylene glycol vapor. In the present invention, the relative humidity should be maintained between 10 to 80 per cent as experiments have demonstrated that little sterilization with glycol takes place at relative humidities below 10% or above 80%. To obtain substantial sterilization of the type desired under most operating conditions, it has been found that a relative humidity of 30% to 50% should be maintained with the 35% to 45% range mentioned above being preferred. Experiments with the above relative humidities have also demonstrated that 0.001 to 0.008 mg. of triethylene glycol per liter of air yields effective sterilization with the range of 0.0025 to 0.005 mg. triethylene glycol being preferred, and that 0.075 to 0.7 mg. of propylene glycol per liter of air yields effective sterilization with the range of 0.1 to 0.3 mg. propylene glycol being preferred.

The operation of the sterilizer in a room or other enclosure will be determined by the characteristics of the enclosure. Thus, observations on the number of air changes per hour, the temperature of the room and the relative humidity must be made for a trial period as well as from time to time as the apparatus is operating. With knowledge of the temperature and relative humidity of the room, the actual weight of water vapor necessary to raise the relative humidity to the desired level may be calculated. When this level has been reached, the quantity of vapors necessary to maintain it will depend upon the infiltration and exfiltration of room air. The introduction of heated, pure glycol and the use of super-saturated, heated glycol vapor creates a fire hazard. However, the addition of water to the glycol progressively decreases this danger as the amount of water contained in the solution increases. Thus, the solutions contained in the reservoir would burn only with great difficulty and the glycol-water vapor emitted from the sterilizer is completely non-explosive and non-inflammable.

The installation of the sterilizer is flexible since it may be placed in the room to be treated or at a distance therefrom and vapors delivered to the room by an air duct. In order to effect thorough mixing of the air and vapors, the sterilizer should be used in conjunction with a fan system. Thus

the vapor could be led directly under a circulating fan with other circulating fans creating air movement or it can be introduced into a duct system and distributed by means of a blower type of fan incorporated into the system.

It is apparent that many different embodiments of this invention may be made without departing from the spirit and scope thereof and therefore it is not intended to be limited except as indicated in the appended claims.

Having thus described our invention, what we claim as new and wish to secure by Letters Patent is:

1. The method of simultaneously effecting substantially instantaneous sterilization of air laden with bacteria or other pathogenic organisms and controlling the relative humidity of the air, comprising bringing air into contact with a mixture of predetermined quantities of glycol and water in vapor form, the relative quantities of glycol and water vapor brought into contact with the air being so proportioned and in amounts sufficient to produce a lethal effect on the air-borne bacteria and to adjust the relative humidity of the air to a point within a range in which said glycol effects a maximum germicidal action.
2. The method of effecting substantially instantaneous sterilization of air laden with bacteria or other pathogenic organisms as set forth in claim 1 characterized in that the glycol is propylene glycol.
3. The method in accordance with claim 1 characterized in that the glycol is propylene glycol introduced into the air in the amount of 0.2 milligram of propylene glycol per liter of air.
4. The method of effecting substantially instantaneous sterilization of air laden with bacteria or other pathogenic organisms as set forth in claim 1 characterized in that the glycol is triethylene glycol.
5. The method in accordance with claim 1 characterized in that the glycol is triethylene glycol introduced into the air in the amount of 0.005 milligram of triethylene glycol per liter of air.
6. The method in accordance with claim 1 characterized in that the quantity of water vapor introduced into the air is so proportioned and in an amount sufficient to maintain said range of relative humidity at from 35%-45%.
7. The method of sterilizing air laden with bacteria or other pathogenic organisms which comprises boiling an aqueous glycol solution to form vapors of glycol and water, the concentration of said solution being such that at the boiling point thereof the vapors in equilibrium with said solution contain glycol vapor in sufficient quantity to sterilize the air, bringing said vapors into contact with the air to be sterilized, and adding water and glycol to said solution as the components thereof are depleted by the formation of vapors to maintain the concentration of said solution substantially constant.
8. The method in accordance with claim 7 characterized in that the aqueous glycol solution is a 95% solution of propylene glycol in water.
9. The method in accordance with claim 7 characterized in that the aqueous glycol solution is a 93.5% solution of triethylene glycol in water.
10. The method of sterilizing air laden with bacteria or other pathogenic organisms which comprises heating a 95% solution of propylene glycol in water to a temperature of from 140°-150° C. to form a vapor containing 25% propylene glycol and 75% water, bringing said vapor into

contact with the air to be sterilized and adding water and propylene glycol to said solution as the components thereof are depleted by the formation of vapors to maintain the concentration of said solution substantially constant.

11. The method of sterilizing air laden with bacteria or other pathogenic organisms which comprises heating a 93.5% solution of triethylene glycol in water to a temperature of not more than 140° C. to form a vapor containing 3% triethylene glycol and 97% water, bringing said vapor into contact with the air to be sterilized and adding water and triethylene glycol to said solution as the components thereof are depleted by the formation of vapors to maintain the concentration of said solution substantially constant.

12. An apparatus for sterilizing air by introducing measured quantities of glycol and water vapor into the atmosphere comprising a covered tank adapted to receive an aqueous glycol solution, means for heating said tank, means for conducting the glycol and water vapor formed in said tank to the atmosphere, means for supplying water to said tank, means for controlling the flow of said water and means in said tank operably responsive to variations in temperature therein to control said water flow control means.

13. An apparatus for sterilizing air by introducing measured quantities of glycol and water vapor into the atmosphere comprising a covered tank adapted to receive an aqueous glycol solution, an electrical heating element mounted in said tank, a vapor duct formed in said tank to conduct vapors to the atmosphere, means for supplying water to said tank, a solenoid operated valve mounted in said water supply means for controlling the flow of water therethrough, a solenoid operably connected to said valve, a thermostat mounted in said tank and an electric circuit including said solenoid and said thermostat, said thermostat being operably responsive to variations in the temperature of the solution in said tank to close said circuit and actuate said solenoid.

14. The method of sterilizing air laden with bacteria and other pathogenic organisms which comprises boiling a solution of propylene glycol and water, bringing air into contact with the vapors generated by said solution and maintaining said solution at a predetermined concentration at which said solution upon boiling generates vapors of propylene glycol and water in quantities so proportioned and sufficient to maintain the relative humidity of the air at from 30% to 50% and the concentration of propylene glycol vapor

in the air at from 0.075 to 0.7 milligram per liter of air.

15. The method in accordance with claim 14 characterized in that said solution is maintained at a predetermined concentration at which said solution upon boiling generates vapors of propylene glycol and water in quantities so proportioned and sufficient to maintain the relative humidity of the air at from 35% to 45% and the concentration of propylene glycol vapor in the air at from 0.1 to 0.3 milligram per liter of air.

16. The method of sterilizing air laden with bacteria and other pathogenic organisms which comprises boiling a solution of triethylene glycol and water, bringing air into contact with the vapors generated by said solution and maintaining said solution at a predetermined concentration at which said solution upon boiling generates vapors of triethylene glycol and water in quantities so proportioned and sufficient to maintain the relative humidity of the air at from 30% to 50% and the concentration of triethylene glycol vapor in the air at from 0.001 to 0.008 milligram per liter of air.

17. The method in accordance with claim 16 characterized in that said solution is maintained at a predetermined concentration at which said solution upon boiling generates vapors of triethylene glycol and water in quantities so proportioned and sufficient to maintain the relative humidity of the air at from 35% to 45% and the concentration of triethylene glycol vapor in the air at from 0.0025 to 0.005 milligram per liter of air.

18. The method in accordance with claim 7 characterized in that the aqueous glycol solution is a solution of propylene glycol in water which upon boiling generates vapors of propylene glycol and water in quantities so proportioned and sufficient to maintain the relative humidity of the air at from 35% to 45% and the concentration of propylene glycol vapor in the air at 0.2 milligram per liter of air.

19. The method in accordance with claim 7 characterized in that the aqueous glycol solution is a solution of triethylene glycol in water which upon boiling generates vapors of triethylene glycol and water in quantities so proportioned and sufficient to maintain the relative humidity of the air at from 35% to 45% and the concentration of triethylene glycol vapor in the air at 0.005 milligram per liter of air.

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