Jan. 2, 1940. J. W. DUDLEY, JR 2,185,940

METHOD OF AND APPARATUS FOR VACUUM CRYSTALLIZING, OR VACUUM COOLING, BY FORCED BOILING

Filed Jan. 24, 1938 2 Sheets-Sheet 1

Fig. 1.

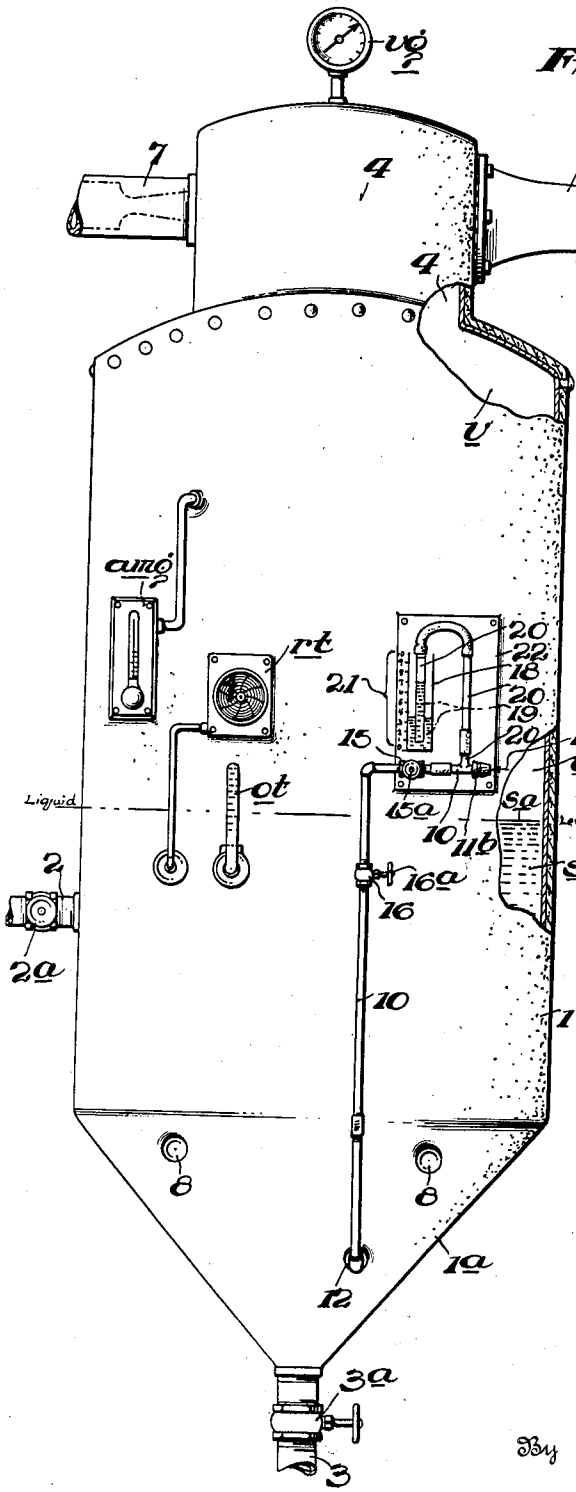

Fig. 4.

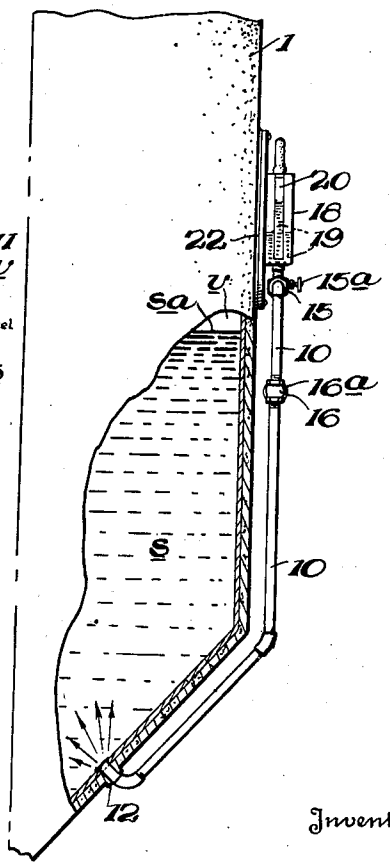

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Fig. 2.

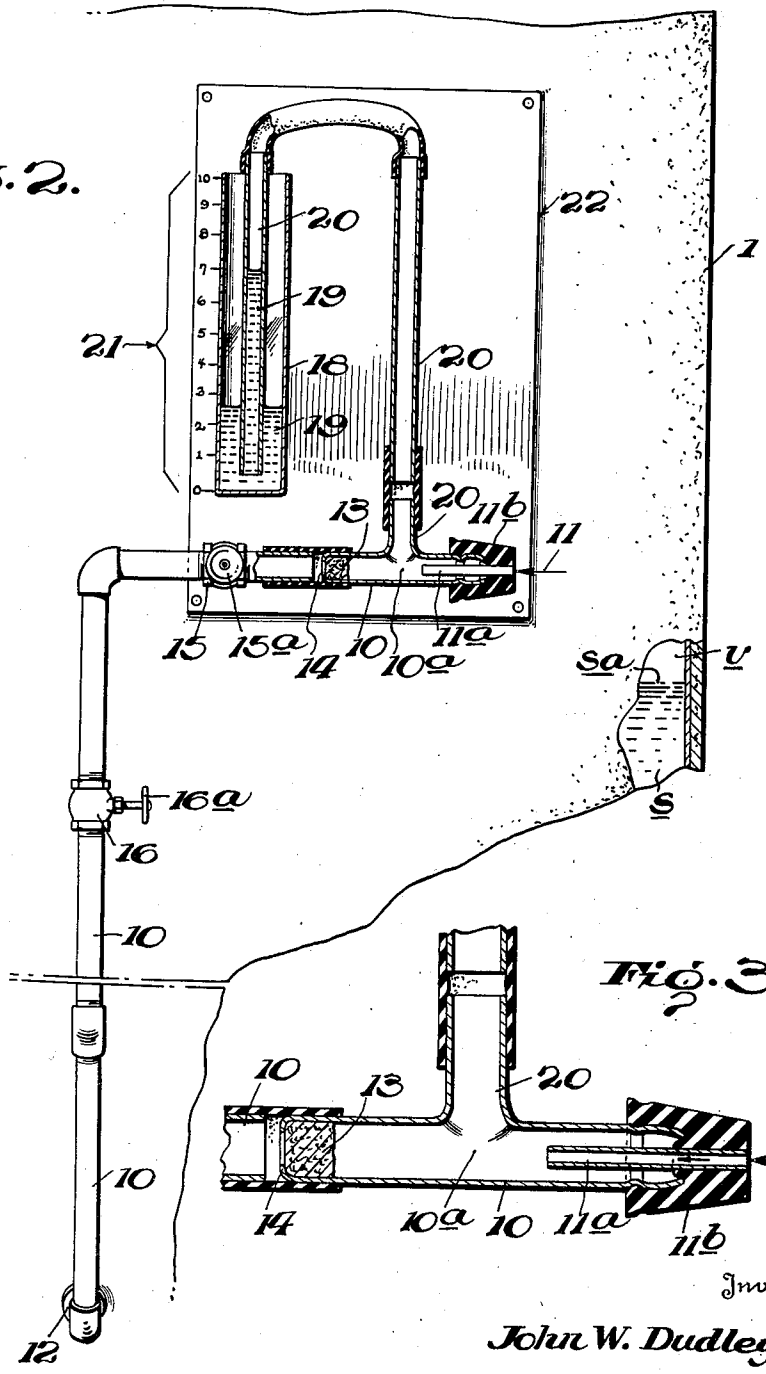

Fig. 3.

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METHOD OF AND APPARATUS FOR VACUUM CRYSTALLIZING, OR VACUUM COOLING, BY FORCED BOILING

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Application January 24, 1938, Serial No. 186,754

15 Claims. (Cl. 62—152)

This invention relates to an apparatus for and a method of vacuum crystallizing, or vacuum cooling, by forced boiling; and the objects and nature of the invention will be understood by those skilled in the art, in the light of the following explanations of the preferred steps following in practicing or carrying out my method, and in the light of the following explanations of the disclosures of the accompanying drawings that illustrate what I now believe to be the preferred embodiment of my apparatus from among other forms, constructions and arrangements within the spirit and scope of the invention.

In that type of vacuum crystallizer, to which my method particularly applies, cooling of the solution and consequent precipitation of the solute in crystallized form, is attained by evaporation promoted by boiling of said liquid forced by low absolute pressure or a high vacuum relative to atmospheric pressure, maintained over the surface of the liquid, and the same also applies to a true vacuum cooler in which my method can be employed, wherein any suitable liquid to be cooled, is reduced in temperature by evaporation promoted by boiling forced by low absolute pressure maintained over the liquid surface.

For example, in a known type of vacuum crystallizer, a high vacuum or low absolute pressure is produced over the solution, by two or more steam jets in series or stages, and/or other suitable evacuating means, operating in conjunction with the vapor offtake from an otherwise closed vapor chamber over the surface of the solution, and leading to a suitable vapor condenser. When the absolute pressure over the liquid is lowered to substantially that of the solvent vapor pressure, the liquid begins to boil, i. e., the liquor actively boils when the ratio of liquor vapor pressure to absolute or total pressure is substantially unity. This boiling removes latent heat of vaporization from the liquid and the liquid hence cools. This cooling is continued to a proper temperature where crystallization and precipitation of the solute takes place, due to the lowered temperature and, also, to partial loss of solvent.

In a true vacuum cooler, broadly, the same action takes place as above outlined, with the exception of crystallization.

It is an object of my invention to improve the efficiency of such vacuum cooling and/or crystallizing methods, by increasing the rate of vapor formation and rate of heat transfer during said liquid boiling stages without thereby substantially or undesirably decreasing the ratio between vapor pressure and absolute pressure over the surface of the liquid.

A further object of the invention is to provide apparatus for vacuum cooling or vacuum crystallizing, by forced boiling, with means for the introduction of a small predetermined and measured flow of inert gas such as air, into the body of liquid being cooled, under manual control or setting, and to provide such means with a suitable indicating gage that will disclose to the attendant, the differential pressure of said inflowing measured air between the air-measuring inlet and the measured air outlet, of said means, said outlet being adapted to discharge into the body of cooling liquid within the cooler or crystallizer and to be subjected to the vacuum therein.

Another object of the invention is to provide a vacuum cooler of the types substantially as described, in combination with means for discharging and controlling measured and predetermined flows of inert gas, such as air, into the cooling liquid under the suction action of the low absolute pressure over the liquor, during the operation of evaporation by forced boiling, and to provide such device with an air passage discharging into said liquor from a measuring air inlet of a fixed capacity at least equal to the maximum mol weight rate of flow required during maximum liquor vapor generation to maintain a predetermined ratio of mol weight air to mol weight liquor vapor substantially constant throughout forced evaporation, by the provision of means to visibly indicate the air pressure differential across said air measuring inlet, and a throttling device for controlling and varying said differential pressure whereby the attendant substantially guided by the liquor temperatures and by said differential pressure indications can control the throttling device to proportionately decrease the mol weight inflow of air as the mol weight of vapor generation decreases, in maintaining said predetermined ratio of air to vapor substantially constant from maximum to minimum rates of vapor generation.

With the foregoing objects in view, as well as other objects that will be developed hereinafter, my invention consists in certain novel method steps, and in certain novel combinations, constructions, and arrangements, as more fully and particularly explained hereinafter and specified by the appended claims.

I have discovered that the rates of vapor formation and heat transfer during said boiling stage, are substantially increased, by the intro-

duction of a small flow of inert gas, such as air, into the body of liquid well below the liquid surface during such boiling, provided that this air inflow is measured and controlled, and bears a certain relation in volume or quantity, to the volume or quantity of liquid or solvent vapor leaving the surface of the liquid body during such boiling. I have also discovered that there is a critical proportional ratio betwen the mol weight rate of air inflow and the mol weight rate of solvent vapor flow, that must be substantially maintained to gain the efficiency sought, and I express this critical proportion, as substantially 1 mol of introduced air per 1000 mols of solvent vapor, within certain hereinafter explained maximum and minimum limits.

The accompanying drawings forming a part hereof, disclose merely as an example, from among other constructions and arrangements that can be employed to practice the instant method, an embodiment of the apparatus invention, from among others within the spirit and scope thereof, for purposes of explanation, and not limitation except where so required by the prior art.

In said accompanying drawings:

Fig. 1 shows in diagrammatical elevation, a form of vacuum cooler equipped with apparatus of the instant invention, portions being broken away.

Fig. 2 is a detail elevation of apparatus of the instant invention for providing a controlled measured flow of inert gas into the liquor being cooled, parts being shown in section or partially broken away.

Fig. 3 is an enlarged detail view of details of said apparatus including an air gage, such as a manometer, parts being broken away.

Fig. 4, shows the air gage in edge elevation and its measured-air discharge open to the liquor in the vacuum cooler tank.

Merely for purposes of convenient explanation of an application of my invention, the vacuum cooler disclosed by the drawings is of the crystallizer type, without desiring to so limit my invention.

The vacuum cooler disclosed, includes an upright tank 1, for the liquor *s*, to be cooled, and, in this particular example, this liquor *s*, is a solution from which the solute is to be precipitated in crystallized form. Where the apparatus is a vacuum cooler for refrigerating purposes, the liquor *s*, is not necessarily a solution.

The tank 1, is supplied with liquor *s*, through liquor supply pipe 2, provided with shut-off and controlling valve 2*a*, whether to periodically replenish the body of liquor in the tank, as in "batch" crystallization, or to continuously feed liquor into the tank to approximately maintain a constant liquor level *sa*, therein, as in "continuous" crystallization.

In the particular example illustrated, the tank bottom 1*a*, centrally and longitudinally contracts downwardly to approximate funnel form and terminates in a depending discharge or offtake pipe 3, for the precipitated solute. This pipe 3, is provided with dump valve 3*a*, and for "continuous" crystallization is liquor sealed at its bottom discharge end and of length to form a barometric leg, although I do not wish to so restrict my invention.

The tank 1, is of the necessary capacity to receive the required body of liquor *s*, and provide an ample vapor space *v*, over the top surface of the liquor *s*.

The tank 1, is provided with any suitable means for evacuating the vapor space *v*, to maintain the desired vacuum (minus pressure) therein and off-flow of vapor and gas or air therefrom. Various means can be provided for this purpose, although in the drawings, I happen to show a closed offtake conduit 4, 5, from the top of the vapor space *v*, provided with an evacuating steam jet stage and venturi 6, supplied through steam pipe and nozzle 7, and leading to auxiliary evacuating means (not shown) and a vapor condenser (not shown) usually provided with a water sealed barometric leg for the water of condensation, as is common in the art, although I do not wish to limit my invention to any particular means for or method of evacuating the vapor space and attaining and/or maintaining the desired vacuum therein or rate of vapor flow therefrom.

The tank 1, and its connections, are preferably designed and fitted to eliminate or prevent air leakage into the liquor *s*, and vapor space *v*, with the end in view of reducing to the minimum or absolutely preventing, if that be possible, accidental or uncontrolled air flow into the tank.

I happen to show, rotary shafts 8, for operating the usual stirrers or agitators located in the lower portion of the body of liquor *s*, for a well known purpose, but my invention is not concerned with the presence or absence of such agitators.

In the example illustrated, a vacuum gage *vg*, is provided to exteriorly indicate the minus pressure within the vapor offtake 4.

A visible absolute mercury gage *amg*, is also provided at the exterior of the tank, and this gage is in operative communication with the vapor space *v*, within the tank, to exteriorly indicate absolute pressure conditions therein above the liquor level.

The tank is also exteriorly provided with an exteriorly indicating or visible thermometer *ot*, in operative communication or direct contact with the upper portion of the liquor *s*, to exteriorly display and indicate the temperature of such liquor.

In the example illustrated, a recording thermometer *rt*, is exteriorly or visibly arranged on the tank 1, and the bulb or temperature responsive portion of this thermometer *rt*, is in direct contact with the liquor in the tank. This recording thermometer, as commonly employed, on such vacuum coolers, is designed to plot a continuous curve of the liquor temperatures over a period of time, during operation, and the operator can hence follow the process of cooling as to proper conduct of the method by consulting the recording thermometer and the absolute gage *amg*.

The vacuum cooler or crystallizer tank 1, and its equipment as thus just described, is in common practice. Whether or not the vacuum cooler tank is equipped with one or more or all of the gages or instruments just described or the equivalent thereof, said tank is, in accordance with my invention, provided with apparatus or means for introducing into the liquor during the boiling stage, a small controlled measured flow of inert gas, preferably air, which means includes a suitable air gage, for instance, a manometer for the guidance of the operator in controlling said air inflow.

Fig. 1 of the drawings diagrammatically discloses, merely as an example, one operative association of an embodiment of such an apparatus or means, with a vacuum cooler tank. Other

figures of the drawings, show details and arrangements of this measured air inflow apparatus, disclosed as an example of one embodiment from among others, for attaining the objects of the invention. In this example, the measured air inflow apparatus provides an airflow line or tube 10, leading from an air-measuring small or capillary inlet orifice, jet or tube 11, of fixed interior dimensions or capacity, arranged to receive inert gas, such as air preferably at atmospheric pressure, and to discharge the flow of air through a measured-air outlet 12, into, preferably, the lower portion of the body of liquor *s*, preferably as near as practicable to the vertical or longitudinal axis of said body, i. e., of the tank 1. This measured-air outlet 12, in this example, extends through the funnel-like bottom 1*a*, of the tank so that the air inflow will pass directly into the liquor for upward distribution in the form of bubbles throughout the same, although I do not wish to so limit all features of my invention. It will be noted that the measured-air discharge or outlet 12, is subjected to the minus-pressure conditions existing in the body of liquor *s*, within the tank 1, and hence the airflow into inlet 11, and through air pipe 10, into the body of liquor through outlet 12, is maintained by the minus pressure existing on the liquor *s*, in tank 1. Consequently, the air flow line 10, is closed or air tight throughout its length to reduce to the minimum possibility of air leakage thereinto, as it is the intent and purpose to deliver a measured predetermined air flow through inlet 11, tube 10, and outlet 12, into the liquor *s*.

The air flow tube 10, is preferably provided with internal means for throttling the air flow, to reduce to the minimum sudden or more or less violent fluctuations in pressure within the tube, bearing in mind that the internal diameter or capacity of the tube 10, is substantially greater than that of the small or capillary inlet 11, open to atmosphere. For instance, I show an air flow throttling air-porous plug 13, held in tube 10, against advancing movement and spaced a substantial distance inwardly from inlet 11, in the direction of air flow toward the measured air outlet 12. In the particular example shown, the tube 10, is provided with an annular internal stop shoulder 14, against which the plug 13, is held by friction and air pressure. This shoulder 14, while constituting a slight contraction in the air passage, does not serve to obstruct the desired air flow. The throttling plug 13, in the example shown, can be composed of a mass of vegetable or other fibres, such as cotton or asbestos fibres, through which the air can be drawn by the minus pressure within the tank. The particular purpose being to throttle the air flow from the space 10*a*, in the tube 10, between the capillary inlet 11, and the air flow throttling device 13. I, also, gain certain advantages by providing a manually-controlled throttling device located in tube 13, spaced behind or inwardly from the plug 13. For this purpose, I locate a manually controlled valve 15, in the tube 10, provided with accessible exterior operating handle 15*a*, by which the interior capacity of the tube can be varied to suit conditions as more fully explained hereinafter. In certain installations, and under certain conditions, this valve 15, can serve the purpose of a cut-off valve to close and open the tube, as well as a throttle valve during operation of the air flow through the tube and into the liquor *s*. Usually, however, I prefer to provide the tube with an emergency shut-off valve 16, having exterior operating handle 16*a*. This emergency shut-off valve, is preferably located in tube 10, between the throttle valve 15, and the tube outlet into liquor *s*.

Usually in small capacity vacuum cooler installations where the air flow is correspondingly small and decided delicacy and accuracy in the manual control thereof are necessary, I prefer to employ a needle valve as the throttle valve 15. In large installations where the air flow through the pipe 10, is correspondingly greater, the same throttling delicacy that can be attained by a needle valve, is not necessary, a rotary plug or other suitable valve can be employed as the throttling valve 15. In some instances, it may be possible to employ a needle throttling valve 15, alone to serve the purpose of the throttling air-porous plug 13, plus the valve 15, but I prefer to employ the two throttling devices in series; namely, plug 13, plus valve 15, as I thereby gain greater accuracy and delicacy in control and less fluctuation on the part of the air or indicating gage. Any suitable visible gage is operatively connected with the space 10*a*, in air flow tube 10, between the measuring air inlet 11, and the air flow throttling means, to disclose the pressure conditions therein. Any suitable vacuum or differential gage can be employed that is of such character and so calibrated as to visibly disclose to the operator the relatively small drop in pressure across the measuring inlet 11, i. e., the difference between the air pressure at the outer side of said inlet and the pressure at the exit end of said inlet within said space 10*a*. Preferably, the scale of the gage is of such character as to indicate or measure in inches (or other unit of length) of liquid head the pressure of the air as it flows through the air-measuring inlet into the space 10*a*, in front of the throttling means, such as the throttling plug, and hence the minus pressure in the pipe line, forward of said plug, is intermediate between the vacuum (minus pressure) in tank 1, and the minus pressure in the pipe line space 10*a*, during operation of the vacuum cooler.

I show a type of manometer, from among other gages and manometers that can be employed. This manometer provides an open-top reservoir 18, for the suitable manometer liquid 19, exposed to atmospheric pressure. The vertical length of the relatively small-diameter minus pressure tube 20, dips a suitable distance centrally into said liquid, and is open at its lower end in said liquid. This tube 20, opens into the air space 10*a*, of the pipe line 10, and extends therefrom to and includes its aforementioned vertical open-end length. This tube is interiorly unobstructed from the space 10*a*, to the manometer liquid and is air tight against air leakage throughout its length and at its junction into pipe line 10. Any suitable manometer liquid of sutable density, for the desired pressure drop, to be measured, can be employed, such as water, mercury, oil, alcohol, etc., each with a scale calibrated accordingly.

Merely as an example, I show water as the manometer fluid, with a corresponding visible verticle scale 21, read by the difference in levels of the liquid in the straight vertical length of the minus pressure tube. Merely as an example for purposes of explanation, and not limitation, this scale is expressed in inches by running progressively upwardly from zero to 10.

In the example illustrated, without intending to

so limit the invention, the manometer and a certain portion of the air-inflow tube **10**, are carried and supported by and arranged on the front face of a suitable panel or vertical supporting frame **22**, arranged exteriorly of and supported in a conveniently visible and accessible position on the tank **1**, with the tube **10**, depending therefrom to its point of entrance into the interior of said tank. In this example, a straight line portion of air flow tube **10**, that includes the capillary inlet **11**, the space **10***a*, and the throttling valve **15**, is arranged transversely across and secured to the supporting panel **22**, with the reservoir **18**, minus pressure tube **20**, and scale arranged above said tube portion.

The tubing **10**, **20**, can be composed of any suitable material with all joints sealed against air leakage, although I happen to show the same composed of glass or other suitable transparent tubing in lengths joined by elastic rubber sleeves against air leakage, and of sufficient stiffness to resist differential exterior air pressures.

The air inlet **11**, is a restricted small or capillary jet or tube, the smallest interior cross section of which forms an orifice, such as a relatively very small inlet orifice through a thin wall, plate or disk, which can be employed instead of a relatively-capillary tube or jet.

Merely as an example, without intending to so limit the invention, I happen to show the restricted air inlet **11**, formed by a relatively very small diameter cylindrical tube **11***a*, open at both ends and extending longitudinally through and carried by a rubber or other contractile or elastic removable plug **11***b*, fitted on the otherwise open inlet end of the relatively large diameter tube **10**, and tightly sealing the same against air leaking except through said tube **11***a*. This removable tube **11***a*, and its sealing plug carrier, permit ready substitution of larger or smaller capacity measured air inlets, where operating conditions or the particular vacuum cooler installation require greater or less air flow. For instance, merely as a non-limiting example for purposes of explanation, the internal diameter of the measured air inlet **11**, formed by tube **11***a*, is about 0.1 inch and the tube **11***a* is in length about 1.5 inches; said dimensions providing a suitable capacity for a batch vacuum crystallizer having a solvent vapor flow of the magnitude of one pound mol of solvent vapor per minute. A suitable manometer fluid to use with said tube inlet, for example, is water.

Some vacuum coolers now in use, of the general type disclosed hereby, are provided with well-known so-called "samplers." Such a sampler usually consists of a well located at the exterior of the tank, open at the top, and provided with a movable cover normally sealing and closing the open top. The interior of such well usually is connected to the interior of the tank below the liquor lever by (*a*) an upper short horizontal connection having a hand valve for opening and closing the same, and (*b*) a lower short horizontal tubular connection also having a valve for opening and closing the same, all so that when necessary for testing, etc., the liquor from the tank can flow into the well. It is possible to utilize this sampler well, when its upper connection into the tank is closed and the lower connection is open, for the discharge of measured air flow into the boiling liquor in the tank, by dipping the discharge end of air flow tube **10**, into the well through the open top thereof, particularly where the discharge end of tube **10** is provided with a closure plug to removably fit in and close the open end of the well, with the discharge end of tube **10**, extending through the plug to discharge the air flow into the well below the plug, permitting the air to flow through the open lower connection, into the boiling liquor *s*, under the action of the suction or minus pressure on the liquor in the tank.

Those skilled in the art are familiar with the method of vacuum crystallization by forced boiling, as now practised in and by crystallizers of the general type illustrated by Fig. 1 of the drawings. The tank **1**, having been charged with the required quantity or "batch" of solvent (liquor *s*), and all inlets into and outlets from the tank (with the exception of offtake **4**) being closed, thereupon, the operation of any suitable vapor, gas and air evacuating means, with which tank **1**, may be equipped, is started to produce and maintain the desired high vacuum (minus pressure) in the vapor space *v*, that promotes vaporization or evaporation of the solvent (liquor) by forced boiling thereof and bubbling and turbulence therein. When the absolute pressure in the space *v*, over the liquor *s*, is substantially reduced to that of the vapor pressure, and liquor begins to boil. This boiling and consequent evaporation, removes latent heat of vaporization from the liquor and the liquor is thereby cooled. The evacuation operation and consequent removal of solvent vapor from space *v*, and cooling of the liquor, is continued to maintain the liquor at the temperature range where crystallization and precipitation of the solute, take place due to the lowered temperature and the partial loss of solvent. Temperatures of about 40° F. or lower, can be attained in this vacuum crystallizing method because absolute pressures in the vicinity of 0.2 inch mercury absolute are maintained in space *v*, at the surface of the solution, with the end in view of producing vapor composed almost entirely of vaporized solvent or vaporized liquor *s*, if not a solution. When this general type vacuum crystallized method is carried on intermittently "batch" by "batch," the temperature of the liquor falls through a definite range to a final low temperature, after which the tank is emptied by dumping the mixture of crystals and mother liquor through the valved pipe **3**, and then refilled with another batch of high temperature solution. When this general type vacuum crystallizing method, is carried on batch by batch, the inert or non-condensable dissolved gases are rapidly exhausted from the liquor or solution during about the first ten or fifteen degrees F. drop in temperature. If the apparatus is then tight against air inflow or leakage, the rate of solution bubbling or ebullition will then slow down to a marked degree. In this batch by batch operation as heretofore practiced crystallization takes place only in a certain low temperature range.

Where this general type vacuum cooling or crystallizing method is carried on continuously, the tank is first loaded with the warm liquor, and then the evacuating step begins to first gradually cool the liquor to a certain optimum temperature just as in batch operation, and thereafter the evacuating means continuously operates, while warm solution is more or less continuously discharged into the tank at a controlled rate to maintain the low temperature of the liquor in the tank substantially constant, and a mixture of crystals and mother liquor is continuously removed through pipe **3**. Where the operation is

thus carried on continuously, the liquor temperature is maintained more or less constant, and the fresh warm solution approximately constantly entering the tank under control of the operator carries in some small uncontrolled amount of dissolved gases which to a slight degree aid in the formation of bubbles and consequent solvent vapor.

At the beginning of the evacuation of the vapor space *v*, the air flow cut-off valve **16**, closes air flow tube **10**, and hence the pressure at the delivery or inner side of capillary inlet **11** (space **10***a*) equals the pressure at the outer or atmosphere side of inlet **11**, and the levels of the manometer liquid in reservoir **18**, and tube **20**, are equal or in a common horizontal plane. Now, the novel steps that my invention introduce into the above described vacuum cooling method are the introduction of a small controlled measured inflow of outside air or other inert gas into the body of liquor *s*, while said liquor is subjected to the vaporizing or evaporating effect due to low absolute pressure existing in space *v*, and is being cooled by the removal therefrom of the latent heat of vaporization, all with the end in view of improving the efficiency of said method by speeding up the rate of vapor formation and heat transfer.

Thus, when the proper and predetermined temperature range of the liquor *s*, has been reached as will be further explained, during the continued operation of evacuating space *v*, of tank **1**, as determined by reading one of said thermometers on tank **1**, the air flow tube **10**, is opened by operation of valve **16**, the throttle or needle valve **15**, being in opened or semi-opened position, thereupon the air flow tube will be opened to inflow of air entering at inlet **11**, and discharging at **12**, into the liquor in the tank, under the suction action of the vacuum or minus pressure over the liquid in the tank. This suction or minus pressure acting on the air flow tube **10**, will create minus pressure in space **10***a*, at the outlet side of inlet **11**, whereas atmospheric pressure exists at the outer or air receiving side of inlet **11**. As the manometer is coupled into said space **10***a*, the manometer reading shows or indicates the minus pressure in said space **10***a*, i. e. the drop in air pressure at the inner or air delivery side of the inlet **11**, and hence the manometer reading indicates the rate of air inflow, and by manipulation of the valve **15**, to increase or decrease its air flow throttling action, this rate of air inflow can be varied to lower or elevate the manometer liquid to any desired indication of the manometer scale, within limits, as conditions within the tank **1**, change and consequently require a decreased or increased rate of air inflow. The effective diameter or capacity of the air flow inlet **11**, is fixed and predetermined, to deliver at least the predetermined maximum mol weight of air per unit of time, and the rate of air inflow through inlet **11**, is determined by the pressure drop or differential into airflow tube space **10***a*, which is indicated by and known from the manometer reading, and hence by manipulation of the manually controlled throttling means, such as any suitable valve in the air inflow tube, any desired predetermined mol weight of air inflow into the liquid *s*, per unit of time, from maximum to minimum, can be attained within certain limits prescribed by the manometer scale.

In the instant example of an embodiment of the invention, the operator in his manipulation of the throttling valve in the air inflow tube **10**, to produce certain variations in the predetermined manometer readings, follows certain variations in the temperatures of the liquid *s*, in the tank, as disclosed by one of said tank thermometers. In thus manipulating the throttle device to attain manometer readings to acccord with temperature changes of the liquor, the operator is guided by an instruction chart prepared by the technician or engineer in charge, to accord to the particular conditions, solution, etc., of the cooling operation to be conducted. This chart will list several liquid temperatures or temperature ranges and for each such temperature will list the particular manometer reading to which the manometer should be brought by manipulation of the throttle device when the corresponding liquid temperature is indicated by the tank thermometer. By various calculations and analyses hereinafter described, and known in the art, I have discovered that certain rates of solvent vapor flow in the tank during the vacuum cooling method, are substantially indicated during normal operation by liquor temperatures shown by the tank thermometer, and that to gain the advantages I seek, there is a critical relation between the volume or rate of air inflow and the volume or rate of solvent vapor flow.

I have discovered, that the addition of a measured amount of air of the order of substantially one mol air per thousand mols solvent vapor, within certain limits, is critical, and this is true regardless of vacuum cooler size and/or capacity, or rate of evaporation and offtake. This ratio of one mol air to one thousand mols solvent vapor is subject to variation about from fifty to two hundred percent, under various conditions, due to different rates of diffusion depending on logarithmic instead of arithmetic changes, as well as on weather conditions. Control of such vacuum operations is affected by hot weather to a greater extent than by cold weather, since available condenser water is much warmer in hot weather, and by other variable conditions.

In the practice of my invention, during the operation of cooling the liquor *s*, air is allowed to continuously flow into the liquor at a measured flow rate through a small air inlet of fixed known capacity, and solvent or liquor *s*, vapor is continuously being produced over the surface of the liquor at a substantially known predetermined rate and volume, and the mixture of air and solvent vapor is continuously being removed from the space *v*, by the evacuating means, and the ratio of the measured air inflow into the solvent or liquor *s*, to the solvent vapor flow, is substantially one mol air to one thousand mols solvent vapor.

The operator, after the preliminary operation of evacuating the vapor space *v*, consults the liquid temperature thermometer on the tank, and then consults his operating chart or other instructions, to note the particular manometer reading called for by the liquor temperature then shown by the thermometer, and then manipulates the air flow throttle valve **15**, to produce in air flow space **10***a*, the pressure called for by said particular manometer reading. For instance, as a mere fictitious example, if the thermometer then disclosed a liquor temperature of, say, 80° to 60° F., the instruction chart might call for an air gage (manometer) reading of 4 to 5 inches, and the operator would thereupon manipulate the air flow throttle valve to decrease or increase the pressure in air flow tube space **10***a*, according to the pressure then existing therein, until the

manometer reading showed from 4 to 5 inches. It would not then be necessary for the operator to change the position of the throttle valve, until the liquor temperature thermometer disclosed a temperature outside of said 80°–60°, range, say a fictitious temperature within a range of 59°–50°. Thereupon by consulting his instruction chart, the operator might find that such liquor temperature, required a purely fictitious manometer reading of say, 3" to 3.5", and hence would then adjust the throttle valve **15**, to bring the manometer to give a reading in said last named range, and so on.

Although the rate of solvent vapor flow changes gradually with changing liquor temperatures, the operator by following his operating chart, in adjusting the air flow throttling valve, will produce air flow into the boiling liquor within the proper air flow solvent vapor flow ratio limits, whether or not the vacuum cooling method is being conducted "continuously" or "batch" by "batch," although in the continuous operation, the liquor circulates at a fairly constant temperature and hence the required manometer reading is also fairly constant, and slightly less inflowing air is required than in the "batch" method. In "batch" operation, the flow of air is preferably started after the liquor has cooled ten to 15° F., below its initial entering temperature, due to the initial removal of dissolved gases.

The flow of solvent vapor from tank space *v*, is not determined by the air inflow measuring means; namely, the small air measuring inlet **11**, of fixed capacity, and the manometer cooperating therewith and showing drop in pressure at the exit side of said inlet.

Said flow of solvent vapor is dependent on various and sundry conditions, but in this example, certain temperatures of the cooling liquor in the tank, have been found to correspond to certain solvent vapor flow in the tank, respectively, under normal operating conditions.

The solvent vapor flow in the space *v*, during the cooling operation, is determined by knowledge of the cooling rate, specific heat and latent heat of vaporization of the liquor, heat of crystallization. By timing the rate of liquor cooling from one degree of the thermometer to the next, the rate of cooling in that temperature range is measured. Knowing the specific heat and latent heat of vaporization of the solvent liquor, and the heat of crystallization of the crystals which are being precipitated per degree of cooling, it is possible to then calculate the pounds of solvent (usually water) which must be evaporated to lower the temperature of the liquor *s*, one degree. Since this solvent is leaving the tank space *v*, as vapor, the vapor flow can then be calculated in pounds per minute. The entire process of vacuum cooling depends on the removal of heat by using it as latent heat of vaporization. If crystallization is not taking place in the particular liquor temperature range under consideration, then the heat of crystallization of the crystals does not enter into the foregoing calculation. The foregoing calculations are made complex by the fact that batch liquor is continually losing solvent and hence weight, and also by the fact that in batch operation, crystallization takes place only in a certain low temperature range. However, the loss of solvent and production of crystals, is satisfactorily measured from time to time by chemical analysis of samples of the liquor or solvent at different points along the temperature cooling range. As long as the vacuum cooler is being supplied with the same or similar quality and analysis of solution liquor for cooling, the foregoing calculations will not vary appreciably from day to day, as is usually the case in industrial practice.

The capacity or smallest internal diameter of the so-called capillary air flow inlet **11**, is calculated by gas-flow formulae to provide at least the maximum air flow, permissible under the approximate ratio of one mol air to one thousand mols solvent vapor, required when the greatest flow of solvent vapor is being produced in the tank, with a corresponding reading of convenient magnitude on the air gage manometer. The highest solvent vapor production in the tank takes place during the higher liquor temperature ranges. Thus, as the liquor temperatures drop, the throttle valve **15**, is adjusted to gradually throttle the inflow of air, to maintain the said approximate air and vapor ratio as the vapor production rate in the tank gradually falls with the falling liquor temperature. The aforesaid ratio of approximately one mol air per one thousand mols solvent vapor was determined at the most critical and unfavorable condition, namely, when the liquor in the tank was cooling at its lowest temperature and the rate of cooling was slowest, and in the light thereof the minimum air flow capacity of the inlet at a convenient manometer reading, and the manual throttle control thereof, below the maximum capacity of said inlet, to meet the reduced air requirements for such most unfavorable tank conditions, was calculated, to give the best results and to maintain said approximate air-vapor ratio, by the use of the manual throttle, over the entire temperature range of the liquor throughout the cooling process.

The volume or quantity of air permitted to enter the liquor *s*, can be changed or varied by adjusting the throttle valve to raise or lower the manometer reading, or by changing the air inlet to one of greater or less fixed diameter or capacity, or by changing the head on the manometer to change the readings, or by changing the manometer fluid to one of a different density.

To indicate the need for throttling the pressure in the air flow tube between the liquor in the tank and space **10***a*, in the tube, into which the air inlet **11** discharges, let it be assumed that the pressure over the liquor in the cooler tank is 0.2 inch mercury absolute and that the barometric pressure is 29.5 inches mercury absolute. Then the vacuum in the tank is 29.3 inches mercury. It is necessary to divide this rather large pressure difference so that a relatively small part of it will produce an air flow of small magnitude through the so-called capillary inlet **11**—**11***a*.

There is a pressure drop from the space above the liquor *s*, through the body of said liquor (due to the head of liquor through which the air bubbles must pass) and the air flow tube **10**, and the open shut-off valve **16**, and then there is substantial pressure drop from open valve **16**, through the throttling valve **15**, and a further substantial pressure drop from the throttling valve **15**, through the throttling plug **13**, and a further comparatively small pressure drop in air tube space **10***a*; namely, in front of the plug **13**, and between the inflowing and outlet sides of capillary inlet **11**, and it is this last named pressure drop that is measured and indicated by the manometer. It is the comparatively large pressure drop through the throttling valve **15** and throttling plug **13**, which must be controlled in order to indirectly control the pressure in tube

space 10*a*, and hence the throttling plug 13, is advantageous in absorbing a substantial proportion of this pressure drop, thereby enabling the operator by adjustments of manually operated needle or other valve 15, to control the pressure drop or value through the said valve 15, to control the pressure in space 10*a*, to produce the desired value reading on the manometer. Whenever the needle valve 15, is adjusted, all of the aforementioned pressure drops are changed, but the only one that must be measured is that through the capillary inlet 11 and 11*a*. Since the vacuum over the liquor in the cooler tank remains substantially constant during an operation, one or two needle valve adjustments, are usually sufficient to control the air flow as long as desired.

The needle valve 15, and the plug 13, are both throttling device and work in unison, and when the two are in series, it is much easier to control by the needle valve and avoid violent fluctuations on the manometer.

I have by experience in the actual practice of my invention, found that, the inflow of a critical measured amount of air into the liquor *s*, during the vacuum cooling thereof by forced boiling, at a point well below the liquor surface, preferably near the central lower portion thereof, attains advantages, as follows:

(*a*) In some instances, a shortening of the over-all cooling time by about ten to fifteen per-cent has been thereby attained.

(*b*) The improvement in the cooling rate by reason of the addition of the proper small inflow of air, is most marked at liquor temperatures below 60° F. Too much admitted air will either ruin or greatly retard the cooling process.

(*c*) The ratio of substantially one mol of admitted air per one thousand mols solvent vapor may appear at first glance, to be insufficient to perform any useful function; I have however, with respect to the practice of my invention in a certain installation, calculated that this small amount of air entering the liquor under high vacuum at a liquor temperature of about forty degrees F. will produce bubbles in the liquor at the rate of approximately four hundred gallons (or approximately 53.5 cubic feet) per minute, due to the very great expansion under the reduced pressure. Hence, any substantially greater quantity of admitted air would fill too large a portion of space *v*, and produce a retarding effect on the flow of solvent vapor from the offtake 4, and tend to disadvantageously decrease the ratio in space *v*, between vapor pressure and absolute pressure, and appreciably slow down cooling of the liquor, while any substantial decrease in the proportion of admitted air, will fail to gain the maximum advantageous result sought.

(*d*) The large volume of expanded bubbles so formed causes an increased area of liquid to be presented from which evaporation or boiling can take place.

(*e*) The upward movement or agitation produced by the air (or inert gas) bubbles will lower the resistance of the vapor-film between any particle of liquor bounded by vapor. That is, the bubbles "stir" the surface and prevent said vapor-film from becoming stagnant.

(*f*) This stagnation without the use of air bubbles is most apt to take place at temperatures below about 55 to 60 degrees Fahrenheit. It is in this lower range below 55 where many crystallization processes must be carried out. Therefore, the air increases the rate of solvent vapor flow, increases the rate of cooling, and increases the rate of crystallization which depends on loss of solvent, and lowering of the temperature.

(*g*) The viscosity of the liquor increases as it cools. The movement of the air bubbles tends to overcome the increasing tendency to stagnation caused by increasing liquor viscosity.

(*h*) If the tank is used for vacuum cooling without crystallization, the air bubbles will in the same way, as above stated, increase the rate of cooling.

(*i*) No extra steam or other power is required by the evacuating means, when the proper amount of air is used. Therefore, the addition of air effects a steam saving due to shorter cooling times, as well as increased output from the apparatus per day.

What I claim is:

1. In that method of vacuum cooling of substantially the types described, wherein evaporation of the liquor is promoted by boiling forced by evacuation of the liquor vapors and air from the space over the liquor; those steps which include flowing air in a predetermined maximum mol weight rate into said liquor during that period of forced boiling when the liquor temperature is in the temperature range of maximum liquor vapor generation, with said maximum air rate controlled and proportioned with respect to said maximum vapor rate generation to maintain a predetermined ratio between mols of air and mols of liquor vapor in said space, and maintaining said predetermined ratio between air mols and liquor vapor mols in said space substantially constant during periods of decreased volume of liquor vapor generation and different liquor temperatures, by proportionately decreasing the airflow rate as the liquor vapor flow rate decreases and the liquor temperature changes.

2. In that method of vacuum cooling wherein boiling and evaporation of the liquor are forced by maintaining low absolute pressure by evacuation of the space over the liquor surface, and wherein the maximum mol weight flow of liquor vapor is generated within a certain liquor temperature range with generation of lesser mol weight flows of said vapor at other liquor temperature ranges; those steps which include increasing liquor evaporation throughout said liquor temperature ranges, by flowing air at a maximum rate into said liquor by the action of said low absolute pressure during said period of maximum liquor vapor generation, in a predetermined controlled minor mol ratio to said maximum mol weight of vapor, and maintaining said ratio of air mols to liquor vapor mols substantially constant in said space through all of said liquor temperature ranges by proportionately decreasing the air inflow rate as the vapor generation rate decreases from maximum to minimum.

3. In that method of vacuum cooling wherein boiling and evaporation of the liquor are forced by maintaining low absolute pressure by evacuation of the space over the liquor surface, and wherein the maximum weight flow of liquor vapor is generated within a certain liquor temperature range with generation of lesser weight flows of said vapor at other liquor temperature ranges; those steps which include increasing liquor evaporation throughout said liquor temperature ranges, by flowing air in maximum weight into said liquor by the action of said low absolute pressure during said period of maximum liquor vapor generation, in predetermined controlled minor mol percentage with respect to said maximum mol weight of

vapor, substantially on the order of one mol inflowing air to one thousand mols liquor vapor; and maintaining said ratio of one mol air to one thousand mols liquor vapor substantially constant in said space through all of said liquor temperature ranges by proportionately decreasing the air inflow rate as the vapor generation rate decreases from maximum to minimum.

4. In the method of vacuum cooling of the types substantially as described, those steps, including, promoting evaporation and cooling of a confined body of liquor by forcing boiling thereof by low absolute pressure maintained by evacuation of the liquor vapors and gases from the space over the surface of the liquor; increasing liquor evaporation during said forced boiling by inflowing inert gas into said liquor under the suction action of said low absolute pressure, and by manual control maintaining substantially constant in said space during said inert gas inflow and liquor vapor generation, a substantially predetermined ratio of gas inflow rate to vapor generation rate on the order of substantially one mol inflowing gas to one thousand mols liquor vapor.

5. In the method of cooling a confined body of liquor; those steps including, promoting evaporation of the liquor by forced boiling induced by low absolute pressure maintained by evacuation of the liquor vapors and gases from the space over the surface of the liquor; increasing liquor evaporation and cooling during said forced boiling, by constantly inflowing air into said liquor under the suction action of said low absolute pressure substantially in a minor predetermined mol weight rate ratio to the mol weight rate of liquor vapor formation, and maintaining substantially constant said predetermined proportion of inflowing air during said forced boiling, at a value less than that necessary to substantially decrease the ratio of vapor pressure to absolute pressure in said space.

6. In the method of cooling a confined body of liquor by evaporation promoted by boiling forced by low absolute pressure maintained by evacuation of the gases and liquor vapors from the space over the liquor surface; including, increasing the rates of liquor vapor formation and heat transfer by inflowing air into said liquor during said forced boiling and liquor vapor formation, under the action of said low absolute pressure, in a substantially predetermined minor mol weight rate proportion to the mol weight rate of vapor formation and maintaining the ratio between vapor pressure and absolute pressure in said space substantially constant throughout said forced boiling, by controlling and proportionately varying said minor air inflow rate as the rate of liquor vapor formation varies.

7. In the method of cooling a confined body of liquor by evaporation promoted by boiling forced by low absolute pressure maintained by evacuation of the gases and liquor vapors from the space over the liquor surface; those steps, including, increasing the rates of liquor vapor formation and heat transfer by inflowing air into said liquor during forced boiling and liquor vapor formation, under the action of said low absolute pressure, in a substantially-predetermined minor mol weight rate proportion to the mol weight rate of vapor formation, by controlling and proportionately varying said air inflow rate as the liquor temperature varies between the temperature range of maximum formation of liquor vapor and the temperature ranges of decreased formation of liquor vapor, in a manner to maintain the ratio between vapor pressure and absolute pressure in said space substantially constant.

8. In the method of vacuum cooling of the types substantially as described; those steps, including promoting the formation of liquor vapor by forcing liquor boiling by low absolute pressure in the space over the liquor surface maintained by evacuation of said space; and increasing the liquor evaporation and cooling rates by flowing inert gas into said liquor by the suction of said low absolute pressure while measuring and controlling said gas inflow to substantially maintain a low predetermined ratio of mol weight rate of inflowing gas to mol weight rate of liquor vapor in said space, substantially constant, and to maintain said proportion of inflowing gas less than the volume necessary to substantially decrease the ratio of vapor pressure to absolute pressure in said space.

9. In that method of vacuum cooling of the types substantially as described, wherein boiling and evaporation of the liquor are forced by maintaining low absolute pressure by evacuation of the space over the liquor surface; those steps which include flowing air under the suction influence of the low pressure in said space, into said liquor during said evaporation, through a measuring orifice of substantially predetermined fixed maximum capacity, and substantially reducing or throttling the pressure of the air inflow between said orifice and said liquor to maintain the air inflow into said liquor in such minor substantially-predetermined mol weight ratio to the mol weight of liquor vapor then being generated as not to substantially decrease the ratio of vapor pressure to absolute pressure in said space; and maintaining said ratio between air flow rate and liquor vapor generation rate in said space substantially constant during liquor vapor generation.

10. In that method of vacuum crystallizing wherein evaporation of the solvent liquor is promoted by boiling forced by low absolute pressure maintined over the surface of the liquor by evacuation of the space over the liquor, and wherein the maximum weight flow of liquor vapor is generated during that period where the liquor temperature is within a certain liquor temperature range, and wherein the weight flow of liquor vapor generated decreases during certain other liquor temperature ranges; those steps which include flowing outside air into said liquor throughout the continuance of said forced evaporation, in volume less than that necessary to substantially decrease the ratio of vapor pressure to absolute pressure in said space; maintaining the maximum rate of inflowing air into said liquor while the maximum weight of liquor vapor is being generated, with said air inflow rate in predetermined controlled minor proportion to said maximum rate of vapor generation; and maintaining said minor proportion of inflowing air to liquor vapor being generated substantially constant during those periods when decreased weights of liquor vapor are being generated, by manually throttling said air flow and correspondingly and proportionately reducing the rate thereof as the liquor vapor rate decreases with changes in liquor temperatures.

11. In that method of vacuum cooling of the types substantially as described, wherein boiling and evaporation of the liquor are forced by maintaining low absolute pressure by evacuation of the space over the liquor surface, and wherein

the maximum weight of liquor vapor is generated within a certain liquor temperature range with reduced generation of such vapor in a different temperature range; those steps which include increasing liquor evaporation by flowing air into said liquor during said liquor temperature ranges by the suction of said low absolute pressure in said space, in controlled predetermined minor mol weight proportion to the varying mol weights of liquor vapor generated during said liquor temperature ranges, with said air inflow less than that necessary to substantially decrease the ratio of vapor pressure to absolute pressure in said space throughout said liquor temperature ranges; and maintaining the predetermined ratio of air mol weight to liquor vapor mol weight in said space substantially constant throughout said liquor temperature ranges by proportionately varying the air inflow rate as the mol weight rate of vapor generation varies.

12. In the method substantially as described, of cooling a confined body of liquor by forced boiling induced by low absolute pressure in the space over the liquor surface maintained by evacuation of the liquor vapors and inert gases from said space, and wherein liquor vapor formation is increased by drawing a flow of inert gas into said liquor by the action of said low absolute pressure, in predetermined minor proportion to the vapor formation rate while the ratio of liquor vapor pressure to absolute pressure in said space is maintained substantially constant, and wherein the maximum rate of generation of vapors occurs during a known indicated liquor temperature range, with a decreasing rate of vapor generation during a different indicated known liquor temperature range, and wherein a predetermined maximum inert gas inflow occurs during a known indicated differential pressure range in said gas inflow, and a predetermined decreased gas inflow occurs during a known and different indicated differential pressure range in said air inflow; those steps that comprise maintaining said minor proportion of inflowing gas to the rate of vapor formation substantially constant during vapor formation by bringing the gas flow differential pressure within said maximum gas flow differential pressure range by throttling said gas inflow, when said liquor temperature is within said temperature range of maximum vapor generation, and bringing said gas inflow differential pressure within said decreased gas flow differential pressure range by manually throttling the gas flow when said liquor temperature is within said liquor temperature range of decreased liquor generation.

13. Apparatus for carrying on the vacuum liquor cooling method of the types substantially described, said apparatus including a vacuum cooler liquor tank provided with means for evacuating the space over the liquor in said tank to force liquor boiling and evaporation by maintaining low absolute pressure in said space, said tank having means providing an inert gas suction inflow passage discharging into the lower portion of the cooling liquor in the tank under the action of said low absolute pressure, said passage including a gas measuring orifice of substantially fixed predetermined maximum gas flow rate capacity; a differential pressure gas gage coupled into said passage to measure the pressure differential across said orifice, said gage provided with a scale including a visible gas pressure differential indication range for maximum gas mol weight rate inflow substantially correlated against the liquor temperature range of maximum mol weight rate of liquor vapor formation, and at least another visible gas pressure differential range for a substantially predetermined proportionately decreased gas mol weight rate of gas inflow substantially correlated against the liquor range of decreased mol weight rate of liquor vapor formation; and a throttle valve controlling the pressure differential in said passage and adjustable according to liquor temperatures to bring said gage to said maximum gas flow reading when the liquor temperature is within said range of maximum vapor formation, and to bring said gage to said proportionately reduced gas flow reading when the liquor temperature is within said range of decreased vapor formation, said passage provided with an air-porous fibrous-mass throttling plug normally-permanently located therein in series with said throttle valve for reducing abnormal pressure differential fluctuations caused by adjustments of said valve.

14. A vacuum cooler structure of the types substantially as described, comprising the combination with a vacuum cooler liquor tank provided with means for evacuating the space over the liquor in said tank to force liquor boiling and evaporation by maintaining low absolute pressure in said space, and wherein the liquor vapor generation rate varies with the liquor temperature, of manually controlled means for inflowing inert gas into said liquor substantially in a minor predetermined mol weight rate ratio to the mol weight rate of liquor vapor generation, and for maintaining substantially constant said predetermined proportion of inflowing gas during periods of maximum and lower mol weight rates of liquor vapor generation, said manually controlled means including an inert gas suction inflow conduit discharging into the lower portion of the liquor in said tank, a gas inlet orifice member communicating with the passage in said conduit and arranged in an elevated position at the tank exterior, said orifice member being of substantially fixed predetermined maximum gas inflow rate capacity, a differential pressure gas gauge coupled into said conduit substantially adjacent to said orifice member to measure the differential pressure in said passage across said orifice member, said gauge provided with a visible scale exposed at said elevated exterior portion of said tank, said scale having markings to indicate changes within a predetermined range in pressure across said orifice member, and a manually controlled variable throttle valve arranged in said conduit between the discharge end thereof and said gauge and accessible at said elevated exterior of said tank, for controlling and varying the gas inflow to bring about a required gauge reading.

15. A structure of the type set forth in claim 14, wherein the orifice member is carried by a friction member removably securing the same to said conduit.

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