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- [73] Assignee **The United States of America as**
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[54] **REFRIGERATING VAPOR BATH**
1 Claim, 2 Drawing Figs.

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62/56
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- [50] Field of Search **62/52, 53,**
50, 51, 216, 217, 169, 268, 100, 208

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ABSTRACT: In the laboratory, or for other limited refrigeration purposes, the cooling effect of a liquid refrigerant may be used for a limited period of time without recompressing and reliquifying the refrigerant. The present disclosure teaches how to accomplish this feat with maximum efficiency by placing the specimen to be cooled in proximity to the liquid, thereby permitting heat transfer directly to the gaseous refrigerant which is vaporizing from the liquid. Further, the rate of cooling is variable, in accordance with the need for cooling, by warming the liquid to vaporize more liquid, thereby increasing the vapor density above the liquid. With greater vapor density (or, we might say, by making the fog thicker) the heat exchange rate (cooling rate) is increased. Conversely, when heat is not supplied to the liquid, and the vapor density is decreased, the rate of heat exchange (cooling rate) is decreased. The rate of cooling, and the minimum temperature obtainable, may be varied further by adjustable pressure relief means to control the "boiling" point of the refrigerant. Vacuum may be applied to the container if desired to lower the boiling point even lower to achieve a lower temperature.

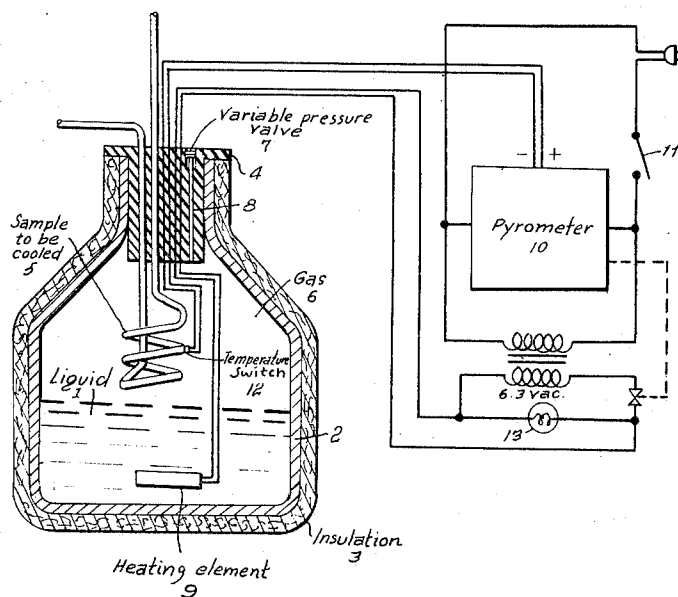


Fig. 2.

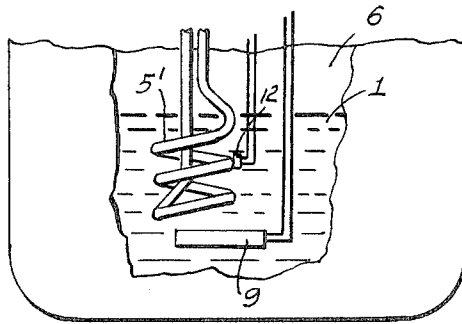
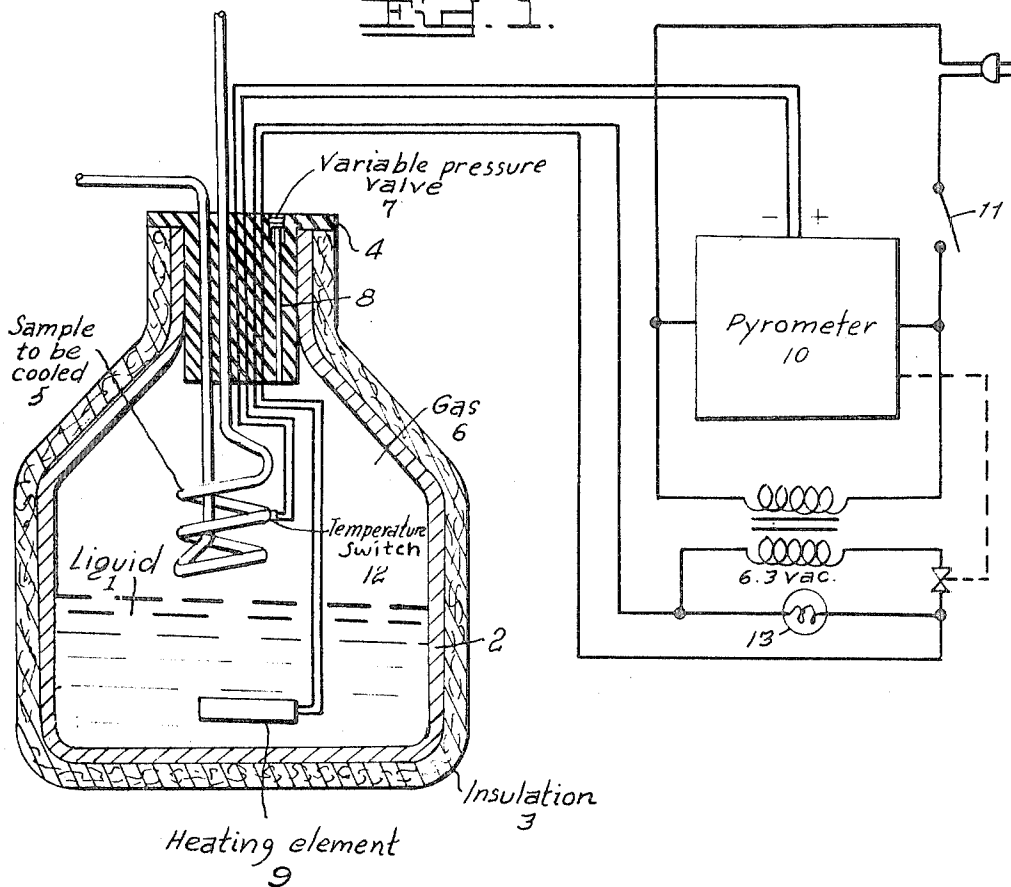


Fig. 1.



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REFRIGERATING VAPOR BATH

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BRIEF SUMMARY

The state-of-the-art method of achieving low stabilized temperatures [below 0° C (32° F.)] in the laboratory has not been significantly advanced in many years. The most practical means available of obtaining known temperatures below 0° C. was to make a slurry of some appropriate chemical with a cooling agent, e.g., dry ice or liquid nitrogen. The cooling agent served to lower the temperature of the chemical to its melting-freezing point, at which temperature it would remain over a wide range of heat transfers.

There are several disadvantages associated with that procedure. First, one must maintain the proper amount of cooling agent in the chemical; if too much is used, all the chemical will freeze, thus lowering the temperature below the transition point; if the cooling agent is not replenished frequently (typically at 1-hour intervals), the chemical will warm above its melting-freezing point and the temperature will increase. Therefore it can become very tedious and time-consuming to maintain a given temperature in a slurry. A second disadvantage is that the temperatures (below 0° C.) are limited to the precise melting points of the available chemicals. Further disadvantages associated with chemical slurries involve fire hazards and toxic vapors.

A technique has been developed recently that is superior in some respects to the one previously described. The sample is cooled by cold-flowing vapor from nitrogen or other refrigerant and the vapor is passed to the sample location through a transfer line.

The advantage in this technique is that the sample temperature can be continuously varied by regulating the flow rate of the cold refrigerant vapor. There are, however, two major disadvantages to this procedure. The first is that even with well-insulated transfer lines a considerable amount of heat loss occurs between the cold nitrogen vapor and the walls of the transfer line. Thus, a large flow rate must be used in order to achieve the required amount of cooling at the sample location. The second disadvantage is also associated with the consumption rate of liquid nitrogen. Due to the heat transfer to the flowing vapor from its environment the vapor is warmed above its vaporization temperature and in this state it is not practical to store the vapor. Therefore one has two options available. The warmed nitrogen vapor may be exhausted from the cooling system, with the requirement that additional nitrogen must be added to replace that which was lost. Or, a refrigeration plant may be employed to cool or compress and cool the warmed vapor to the liquid form so that it can be returned to the liquid nitrogen reservoir. The first choice is not desirable because it leads to a very large consumption rate of liquid nitrogen. For example, on a simple system it was found that about 1.5 liters per hour of liquid nitrogen were required to maintain a sample temperature of -160° C. The alternative is impractical in most laboratories because of the economics associated with purchasing and operation of an adequate (high pressure) refrigeration plant. For the above reasons, the flowing vapor system described above is seldom employed for cooling purposes.

The stationary vapor bath described hereinafter was devised to reduce the consumption rate of liquid nitrogen and, hence, make the use of vapor cooling more practical. When a comparison was made between the consumption rate of liquid nitrogen in the present system and that of a flowing vapor system it was found that the present system was more than 25 times as efficient. The high efficiency is achieved through the elimination of a vapor transfer line and other features as will become more evident hereinafter. Furthermore, the dangers from fire hazards and toxic vapors are negligible in this system. IN THE DRAWING

FIG. 1 illustrates the principles of the invention;

FIG. 2 illustrates a modification.

A charge of liquid refrigerant 1 (e.g., nitrogen or other refrigerant) is placed in container 2 preferably surrounded by insulation 3 and closed by a member 4. A material to be refrigerated, a gas for example, may be passed continuously through tubing 5, or allowed to remain for a period of time and then removed in its refrigerated condition. Of course, bulk material, solid articles or other items to be cooled could be placed inside of container 2 above liquid 1.

The liquid refrigerant begins to boil or vaporize and forms a gas or vapor 6 above the liquid. Adjustable valve 7 is provided in passageway 8 leading from the interior of container 2 to the atmosphere.

A heating device 9 may be provided in proximity to or directly in the liquid refrigerant 1. The heating device may be energized by electricity, for example, introduced through a known type of pyrometer 10 controlled by manual switch 11 and automatic temperature switch 12. An indicator telltale lamp 13 lets the attendant know when the apparatus is energized. The electrical apparatus is of a type well known to those skilled in the art. It may be of the on-off type or of the variable supply type whereby the rate of heat supply may be varied to obtain differing heating-cooling rates.

OPERATION

A charge of liquid refrigerant 1 is placed in container 2 and the closure member 4 is inserted. (If it is desired to contain high bursting pressures the container, closure member, fittings and so on will be appropriately designed.) If very low temperatures are to be produced, the refrigerant may be liquid nitrogen or such with a very low boiling point. If intermediate "cold" temperatures are desired a refrigerant with a higher boiling point may be used, e.g., R-12, boiling point -21.6° F. at atmospheric pressure. If the "cooling" is to be from "hot" (e.g., 212° F.) to "warm" e.g., 100° F.), R-11 could be used, boiling point 74.7° F.

Valve 7 may be used to bleed off air originally trapped inside and the valve may then be set for a desired pressure buildup. After that pressure is reached valve 7 will release gas to maintain the preselected pressure. Or, the valve may be completely closed to allow the pressure to rise to its maximum value for the particular liquid gas and the ambient temperature, e.g., R-12 will rise to a pressure of 77 lbs./sq. in. at an ambient "room" temperature of 75° F. This would extinguish the cooling action and preserve the refrigerant until further cooling is desired at which time the pressure would be partially or completely released. Or, valve 7 may be opened to permit the liquid to boil or vaporize at its lowest temperature at the prevailing ambient or atmospheric pressure; e.g., R-11 boils at atmospheric pressure (14.7 lbs./sq. in. absolute) when at a "room" temperature of 74.7° F.

The gas 6 will be at approximately the same temperature as the boiling liquid. Therefore, almost any desired temperature of gas 6 may be obtained by selecting a refrigerant with a boiling point lower than the temperature sought, and by permitting pressure to escape via valve 7 to maintain that pressure on the chosen refrigerant which corresponds with the temperature desired. If desired, a partial vacuum may be applied to container 2 in order to achieve an even lower temperature with a particular liquid.

SOLUTION TO THE PROBLEM

As explained above, various refrigerants may be chosen for different sets of temperature conditions in laboratory equipment of this type. However, the cooling rate with a given refrigerant, under given fixed conditions of pressure, ambient temperature, insulation and so on, was not variable under prior teachings. In the present invention the rate of cooling is variable merely by adding heat to the liquid; or by adjusting the pressure in the container, or both. A faster cooling rate is obtained by adding heat. Prior to turning on heating element 9 liquid 1 boils or vaporizes to produce gas 6 to a point of equilibrium or a saturated state of the gas above the liquid. Further evaporation of the liquid is equaled by reentry of

some of the gas back into the pool of liquid. The liquid and gas are at the same temperature, that is, the boiling temperature of the liquid for the surrounding pressure conditions (e.g., at atmospheric pressure, approximately: 212° F. for water; 117° F. for R-113; 75° F. for R-11; 38° F. for R-114; -22° F. for R-12; -41° F. for R-22; -115° F. for R-13; and -319° F. for nitrogen).

Adding heat to the liquid produces only minor warming of the liquid and gas and a pressure buildup in the container. Therefore, the principal effect of the additional heat is that the liquid vaporizes faster and adds more of the relatively "cold steam" or "fog" to the gas space. For example, at liquid nitrogen temperature of -196° C. a temperature change of approximately 3.5 percent will change the vapor pressure by 100 percent. More specifically, in a system of fixed geometry, the rate of heat transfer, dQ/dT , from the sample to the refrigerant is proportional to the vapor pressure, P , of the refrigerant and the difference between the sample temperature, T_s , and the refrigerant temperature, T_R . Hence,

$$\frac{dQ}{dT} \propto P(T_s - T_R)$$

Thus a 3 percent increase in refrigerant temperature will increase the lower temperature limit of the sample by the same amount, but the rate of sample cooling at intermediate temperatures will be increased by approximately 100 percent. Expressed in other words, the rate of heat transfer is increased between the gas and the item being cooled and the cooling action is accentuated despite the slight increase in temperature of the gas.

By way of contrast, previous systems increased the rate of heat transfer by flowing or "blowing" more of the vapor around the item to be cooled. This required much more of the vapor and used much more liquid to accomplish the desired cooling. The vapor in contact with the liquid was not saturated and therefore there was not as much reentry of the gas to the liquid as in the present system when the vapor is saturated. In the present system much of the saturated vapor returns to the liquid even while heat is being supplied.

In a modification, illustrated in FIG. 2, the sample 5' is placed in the liquid refrigerant and the sample temperature is regulated directly with the liquid refrigerant temperature which in turn is controlled through evaporative cooling. For example, the liquid refrigerant temperature will rise to the value characteristic of the refrigerant at the given pressure within the container. Therefore the liquid and the sample temperature can be controlled by regulation of the vapor pressure thereabove by means of valve 8. Almost any desired temperature of the sample may be obtained by selecting a refrigerant with a boiling point lower than the temperature sought and by regulating the rate of vapor release. It may be noted that the heat originally contained in the sample to be cooled causes a portion of the liquid to evaporate and the evaporating liquid, in turn, helps cool the sample. Thus heat is conserved in this modification thereby reducing the requirement for heat from the heating element 9.

THE METHOD

The method of cooling embodies the following:

Pressure inside of the container is normally held above atmospheric pressure. This keeps the vapor pressure higher and keeps the "fog" or vapor thicker thereby increasing the rate of heat transfer or cooling of the item being cooled. The adjustable pressure release valve is set to determine the vapor pressure and density of the vapor for the desired cooling rate. A higher cooling rate is obtained as heat is added, the pressure is increased, and the vapor becomes thicker. However, if it is necessary to obtain the lowest possible temperatures, the pressure must be lowered to permit the liquid to evaporate more freely at a lower temperature.

A two-step or a three-step method may be followed. First, the pressure release valve may be set for a relatively high vapor pressure with consequent high vapor density and rapid cooldown of the item. Secondly, after the initial cooldown or rapid prechilling, the pressure may be lowered toward, or to, atmospheric pressure. This permits the liquid to boil at a lower temperature level (say -196° C. for liquid nitrogen at atmospheric pressure) and therefore yields colder liquid and vapor. Therefore, in either FIG. 1 or FIG. 2 the item can be cooled to a very low temperature. If an even lower temperature is required, as a third step, a vacuum can be applied to the container to achieve a lower boiling point of the liquid with a lower consequent temperature of the liquid and vapor and the item being cooled.

Thermostat or temperature sensor 12, and control 10 associated therewith, may be adjustable to supply more heat when greater cooling is desired, or less heat when less cooling is desired. The net result, as pointed out hereinabove, is that the present system is more than 25 times as efficient as a flowing vapor system tested. Thus, the present apparatus and method of operation are very simple, low in cost, long lived, easy to maintain and highly efficient. The rate of cooling is controlled by very simple apparatus which simply adds a small amount of heat as necessary to increase vapor density above the liquid.

We claim:

1. Apparatus for refrigerating an item comprising a container in which the item is to be placed, liquid and gas refrigerant in said container in close proximity to each other, means to produce heating of the liquid to increase the vapor density adjacent to the liquid and surrounding the item to be cooled to thereby increase the rate of cooling, means automatically responsive to an increase in the temperature of the item being cooled to increase the temperature of the heat-producing means to thereby increase the cooling rate, pressure release means to limit pressure buildup in said container, said pressure release means being adjustable whereby the pressure and boiling point of the liquid may be raised to thereby raise the vapor density and increase the cooling rate while conserving cooling liquid and vapor, and said pressure release means being completely closable to stop all loss of vapor and liquid from said container.

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