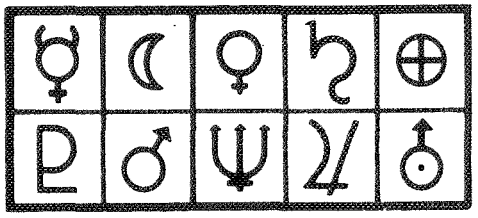

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A PRECISELY CONTROLLED, LOW RANGE HUMIDITY SYSTEM

Daniel M. Garst
Kermit F. Lindell
Planetary Quarantine
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ABSTRACT

Controlled humidity systems had been developed for studies relating the effect of relative humidity to the dry heat inactivation of microorganisms. This report describes an extension of this development in which very low relative humidity values were obtained by pressurizing the saturation portion of the system. Even lower values were attained by subsequently passing the air through a desiccant bed. A discussion of the pressurization principles is included.

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A Precisely Controlled, Low Range Humidity System

Introduction

The relative humidity (RH) of air is of interest in dry heat sterilization studies because it has a definite effect on the heat sensitivity of microorganisms. This relationship has been demonstrated using the NASA standard test organism, Bacillus subtilis var. niger. It was found that the 105° C dry heat D value[1] changed from 2.3 hours to 5.3 hours when the RH was varied from 20 to 60 percent.[2]

Two humidity systems were developed[3] which provide air with closely controlled RH for both dry heat and thermoradiation[4] studies at Sandia Laboratories. These systems are capable of providing a continuous supply of air with an RH in the 20 to 60 percent range at 26° C, within ±1 percent of the desired value.

It has been postulated by mathematical models[5] that at some point below 10 percent RH, the heat resistance of bacterial spores no longer decreases, but begins to increase again. Preliminary tests indicated this point might be less than 1 percent RH at 105° C. In an effort to locate the point at which this increase begins, or conversely, the point in this region at which heat resistance is lowest, it was necessary to reduce the RH furnished by the humidity system. For this reason, the original system used for dry heat studies was modified to provide the needed low-range RH capability. This report describes these modifications and discusses the results attained.

Original Humidity Control System

In the original humidity control system[3] developed for spacecraft dry heat sterilization studies, the RH of the air was controlled by controlling the temperature at which the air was saturated. For example, if air is saturated at 3° C, the RH at 26° C and 105° C is about 22.5 percent and 0.63 percent, respectively (Figure 1).

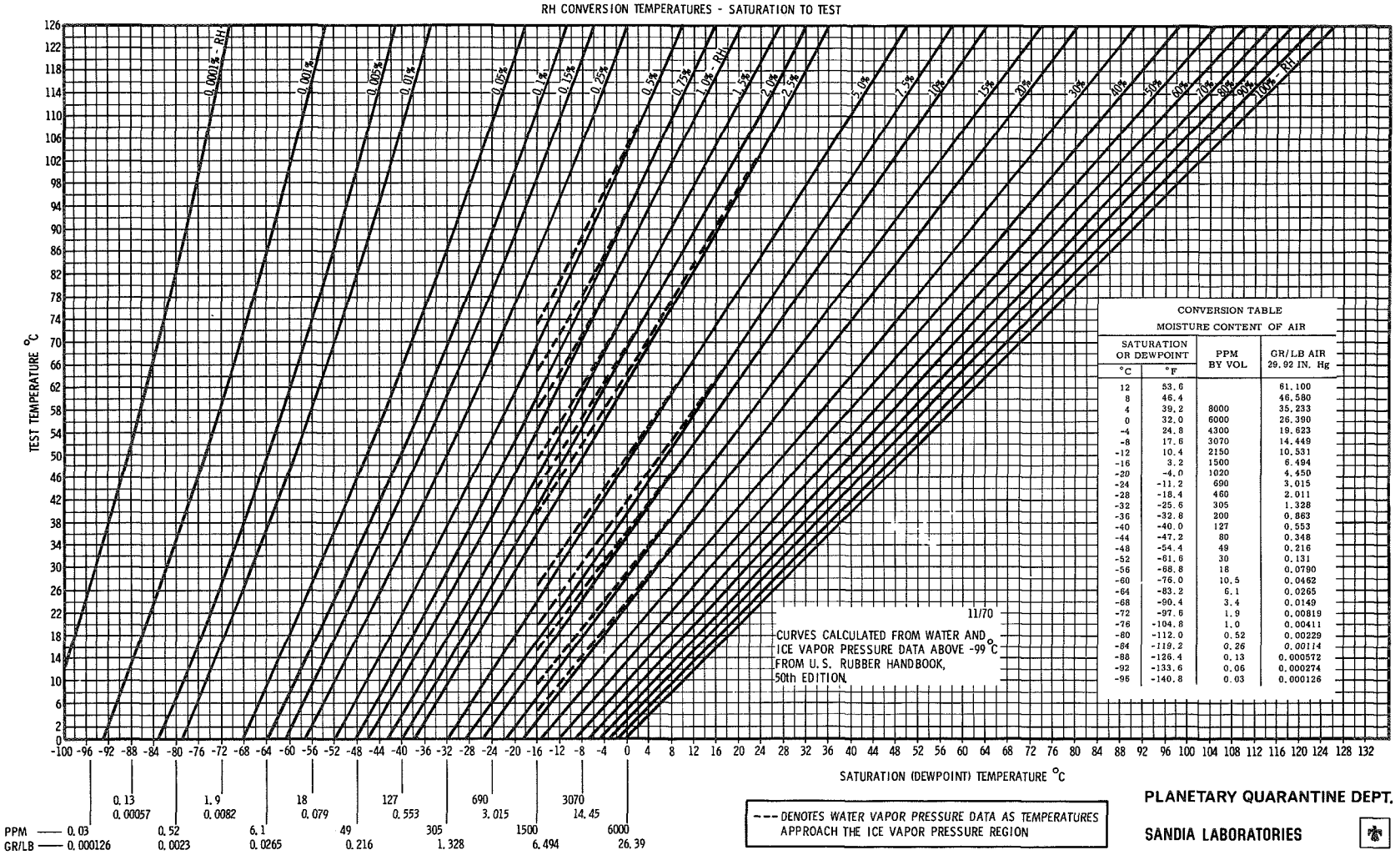

Figure 1. Temperatures of Relative Humidity Conversion

Higher RH values could be attained by increasing the saturation temperature. These calculated values were verified by calibrated lithium chloride (LiCl) specific range humidity sensing elements and recorders. As even lower RH values were needed to explore moisture effects on microbial inactivation in the "very dry" region, other means were developed to meet this requirement.

The original system, using an airflow of approximately 1 cfm, operated at virtually ambient pressure of 12.2 psia. Another consideration was the fact that 2°C was about the lowest practical saturation temperature since excess moisture condensing within the system tubing as well as water in the cold bath would freeze as the temperature approached 0°C. In order to override this limitation and yet obtain lower RH values, we decided to pressurize the system through the point of saturation.

Pressurized Humidity Control System

Effect of Pressure

The operation of the pressurized system is based on the assumption that water vapor and air act as ideal gases and therefore ideal gas laws apply. Under these conditions, one can use the accepted formula for relative humidity[6]

$$\%RH = \frac{e}{e_{sat}} \times 100$$

and add provisions for the variation in pressure to derive the formula

$$\%RH = \frac{e \cdot \frac{p_1}{p_2}}{e_{sat}} \times 100$$

where

e = vapor pressure of air at the saturation temperature

e_{sat} = saturation vapor pressure of air at the temperature used for measuring RH

p_1 = ambient air pressure absolute

p_2 = air pressure absolute at the point of saturation

Two parameters of the system may be varied to achieve innumerable RH values, particularly in the lower RH range. The saturation temperature of the cold water bath can be regulated to provide accurate control down to 22.5 percent RH at 26°C. The addition of pressure to the system increases its capability in the region below 0.6% RH at 105°C. The pressure, in effect, acts as a "vernier" control to further reduce the RH in fractional increments. Using the formula noted above and assuming the following conditions which are typical, we can show the effect of pressure in the following example:

3°C saturation temperature (vapor pressure in mb = 7.575)

26°C measurement temperature (vapor pressure in mb = 33.608)

12.2 psia

73.2 psia in the system

The formula now reads

$$\%\,RH = \frac{7.575 \cdot \frac{12.2}{73.2}}{33.608} \times 100$$

or

$$\%\,RH = 3.75 \text{ at } 26^\circ C$$

The corresponding RH at 105°C would then be 0.105%.

There does appear to be a practical limit beyond which increasing the saturation pressure yields only a marginal reduction in RH. Figure 2 illustrates this relationship. This limit appears to be about 5 atmospheres. Further increasing the pressure in increments of one atmosphere produces only slight additional RH reductions when the air is expanded to ambient pressure.

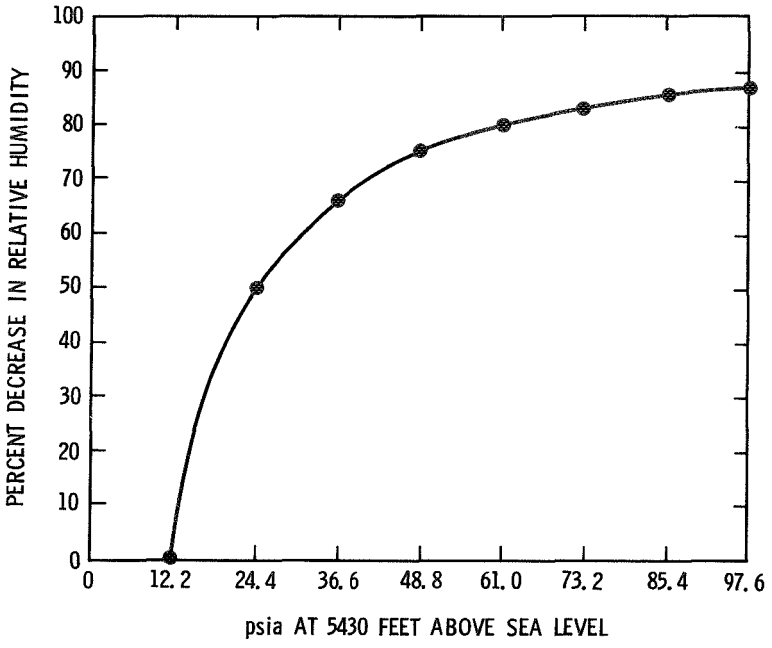

Figure 2. Saturation Pressure Effect on Relative Humidity

Pressurized System Design and Operation

The items of laboratory equipment comprising the pressurized humidity control system are shown in Figure 3. This system is similar to the original system concept, but it differs in several respects. The basic difference is the addition of pressure up to the point at which the valve is shown in the schematic. This change necessitated replacing the chambers in the warm and cold water baths with pressure vessels, replacing plastic tubing with copper tubing, and adding the pressure gage and valve. The position of the flow meter was also changed to remove it from the pressurized portion of the system and all other existing lines were replaced with copper tubing to eliminate any diffusion of ambient moisture into the system through the walls of the plastic tubing. Air from a central pressurized supply enters the system through a pressure regulator. In order to attain both the desired pressure within the system and the desired flow rate of air into the temperature chamber, all adjustments affecting pressure are made concurrently due to the interdependence of their effects.

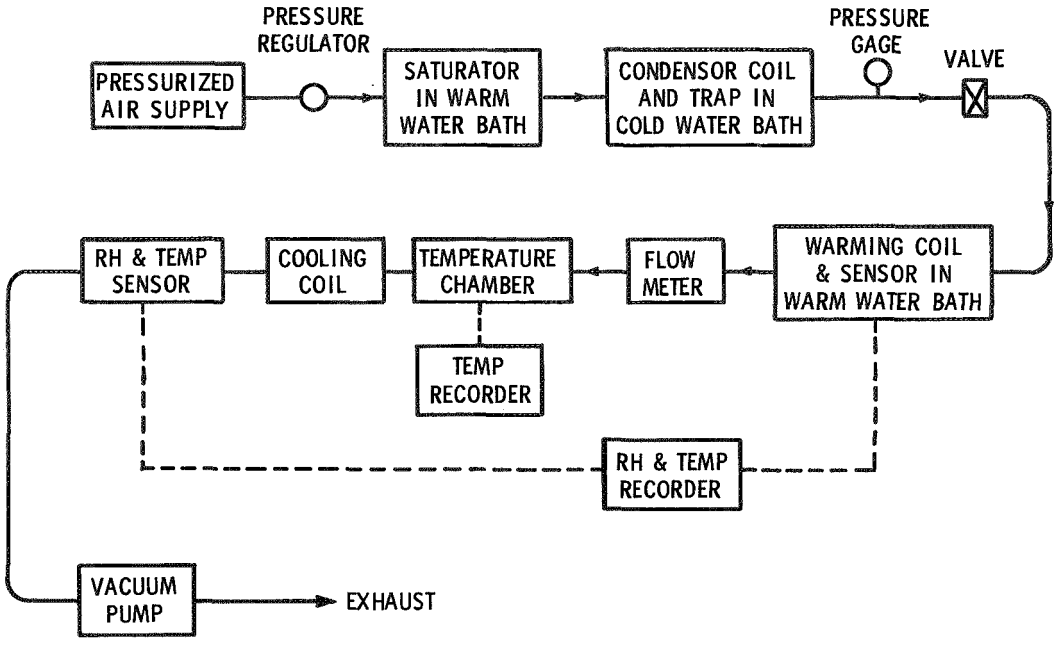

Figure 3. Pressurized Humidity System

The air is then directed through fritted glass gas dispersion tubes submerged in a pressure vessel which is located in a constant temperature (26° C) water bath. This bath temperature was selected because it is slightly above room ambient and therefore is not subject to minor variations in room temperature. The air then proceeds through coils in a cold constant temperature bath where complete saturation is achieved when its temperature is reduced. Excess moisture is condensed and collected in a pressurized trap at the bottom of the coil. This condensate can be expelled periodically through a valve and tube arrangement, using system pressure. For most of the low range RH studies, the cold bath temperature is maintained at 3° C ± 0.1° C. At this point in the airflow, the system pressure is measured and the desired amount of air, usually 1-2 cfm, is metered through a valve. As the air passes through the valve, it is expanded to one atmosphere pressure and the calculated reduction in RH occurs.

The air is warmed again to 26° C, the temperature and RH are measured and recorded, the flow rate is measured, and the air is introduced into the temperature chamber where microbial inactivation experiments are conducted. A continuous air sample is withdrawn from the temperature chamber and cooled to ambient temperature, and the temperature and RH are again measured and recorded. Temperature and RH measurements are made with LiCl specific range sensors and multipoint strip chart recorders which are calibrated as a system by Sandia's Primary Standards Laboratory. As a result, RH measurements are accurate to ±1 percent at ambient conditions.

Modified Pressure System with Desiccant Bed

While the pressurized humidity system extended the low range RH capability beyond that of the original system, still lower RH values were desired for the dry heat experiments. In subsequent modifications, the pressure aspect was retained, but no attempt was made to saturate the incoming air. Components of the pressurized system were used as much as possible for convenience even though in some instances they were not essential to the proper functioning of the modified system.

System Design and Modification

As shown in Figure 4, air enters the system through a regulator from the building compressed air supply at the desired pressure and is cooled as it passes through the coils in the cold water bath. Excess moisture is thereby condensed out of the air. At this point the air is expanded to ambient pressure, warmed, and fed through a desiccant bed to the temperature chamber.

The desiccant chamber consists of an air-tight container about 3 ft^3 in volume. About 6 inches of desiccant ($CaSo_4$) are supported in the center of the chamber by a false, porous bottom. The air enters the chamber into the plenum created by the false bottom, passes up through the desiccant bed, and exits through an air line near the top of the chamber.

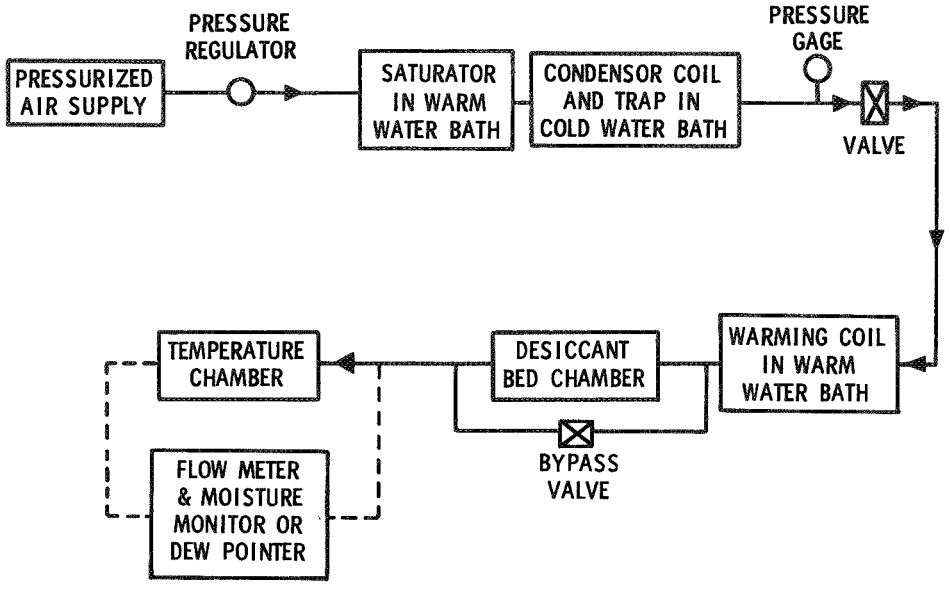

Figure 4. Pressure-Desiccant Humidity System

An essential feature of the desiccant chamber is the bypass arrangement. Without this feature, there is a gap in the RH that can be attained between the lowest practical setting for the pressurized system and the one lower RH value provided when the entire airstream passes through the desiccant bed. By regulating the amount of air passing through the bypass valve, any desired value down to the full capability of the desiccant bed can be achieved.

It should be noted that LiCl sensors are not used in this system because the humidity values are below the low limit of the lowest range sensor available. Therefore, either moisture monitors or dewpointers are used to measure the moisture content of the air. As indicated in Figure 4, these instruments may be located so as to extract a sample of the air entering the temperature chamber or a sample of the air directly from the temperature chamber or both. The readings obtained at these two locations will be virtually identical if there is no induction of ambient air into the temperature chamber and a slight overpressure is maintained. When a moisture monitor is used, the readings in parts per million are then converted to percent RH at the desired temperature, such as

$$\%\,RH = ppm\ (3.31 \times 10^{-3}) \text{ at } 26^{\circ}C$$

or

$$\%RH = ppm\ (1.14 \times 10^{-4})\ \text{at}\ 105^{\circ}C.$$

When a dewpointer is used, the percent RH is readily determined by using the chart in Figure 1, following the dewpoint (same as saturation temperature) line to the temperature of interest, and observing the relation of that point to the RH curves.

During these studies, it was found that the following conditions affect the accuracy of low range RH measurements by some electronic devices.

Instrument Accuracy -- The measuring instrument itself should be calibrated against a reliable standard to establish its inherent accuracy or to permit compensation for the degree of known inaccuracy.

Flow Rate -- These instruments are usually designed for use with a precise flow rate of air through the instrument. Variations in the flow rate will usually result in measurement inaccuracies.

Equilibration Time -- The time required to equilibrate an instrument to very dry test conditions may vary from several hours to several weeks, depending on the prior humidity conditions to which it had been exposed. Purging the instrument with dry nitrogen prior to use can greatly reduce the equilibration time.

Pressure Drop -- Care should be taken to assure that there is virtually no pressure drop of air flowing through the instrument and particularly through the sensing element. Otherwise, erroneous meter readings may result.

The point to be made here is that the operator must be thoroughly familiar with the moisture measuring instrument and its mode of operation in order to obtain accurate results. Most of these instruments have direct reading meters in ppm and some have scale multipliers which further increases the ability of the operator to read them accurately. By comparison, the dewpointer is a much less complex apparatus which will provide accurate and repeatable results. However, it too should be used by an experienced operator.

The other element of the modified system that requires periodic monitoring is the anhydrous condition of the desiccant itself. The desiccant bed that we are using consists of about 35 pounds of $CaSo_4$. It has been used intermittently for a period of 4 weeks and at this point shows negligible degradation of its moisture absorbing capacity. This may be due in part to the fact that both inlet and outlet connections are capped when it is not in use. The entire bed will be replaced with new desiccant when any significant degradation is noted.

Results

The original humidity control system verified the premise that RH could be predictably and reliably controlled by controlling the temperature at which air is completely saturated. The addition of pressure to the system extended the low range RH capability down to about 16% of that attainable without pressure. Thus, at any selected saturation temperature, pressurization of the system in increments of 1 atmosphere provides in effect a vernier control to further reduce RH. For example, an RH of 23% at 26° C can be reduced to 3.75% by the addition of 5 atmospheres pressure over ambient. Step by step reductions in RH were verified by the use of calibrated, specific range, LiCl humidity sensors and dewpoint measurements.

Even further reductions in RH were made possible by modifying the pressurized system such that the saturator was bypassed and the air was directed through a desiccant bed after expansion to ambient pressure. In this drier system, a continuous supply of air with a moisture content as low as 40-50 ppm was achieved. Converted to RH, these values represent 0.132-0.166% RH at 26° C. With the addition of controllable bypass arrangement around the desiccant bed, any RH value between 0.132% and 3.75% may be selected and maintained.

Conclusions

The pressurized humidity control system makes possible a constant supply of air with an RH in the relatively low ranges and provides a direct method for controlling the RH in the environment surrounding microorganisms during dry heat sterilization. The use of a desiccant bed in conjunction with pressure further reduces the RH to the low ppm range.

While closed systems can provide similar relative humidities, the effect on RH of oxide layers and monolayers of moisture within the system probably has not been determined. Depending on the RH of the air in a closed system, a supersaturated condition may exist during heat up, which can bias the experimental results. A closed system may also present subtile problems with regard to pressure affecting the RH.

The principal advantage of the system described in this report is that it is an open, "flow-through" system. Pressure is used only to attain the desired RH and is not present in the dry heat environment. Any moisture driven off the experimental samples is quickly removed by the flow of air through the temperature chamber. And finally, the experimental samples may be quickly and easily inserted or removed from the temperature chamber with virtually no effect on the temperature or RH within the chamber. This feature substantially reduces the duration of heat up and cool down periods.

Notes and References

1. A "D value" is the time required for a viable microbial population to be reduced by 90 percent or 1 log at a given temperature.

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4. Thermoradiation is defined as the simultaneous exposure to heat and gamma radiation.

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