

TRUST BUT VERIFY – PRACTICAL APPROACHES TO HUMIDITY GENERATION AND MEASUREMENT

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ABSTRACT

Construction of humidity generators is a common undertaking in many of today's national metrology institutes and commercial calibration laboratories. A generator based on theoretical methods and statistical uncertainty expectations is often trusted as a laboratory's primary humidity reference. While it is important to follow sound design theory, and compute statistical estimates of the output from a generator, verification is an equally powerful tool that can help to make even the weakest of designs more trustworthy. Generator designs will be reviewed in an effort to avoid common obstacles while capitalizing on a few simple and practical improvements. Testing and verification will also be considered with a focus on condensation hygrometry.

1. INTRODUCTION

In 1948, Weaver and Riley at the National Bureau of Standards (now currently known as the National Institute of Standards and Technology, NIST) developed a 'pressure method' used for the generation and control of humidity. Their method did not rely on measuring the amount of water vapor, but rather was derived from the measurements of temperature and pressure. Termed the "two-pressure principle", this method involved saturation of air, or some other gas, with water vapor at high pressure and then expanding the gas to a lower pressure. If saturation and expansion were performed under constant temperature conditions, the resulting Relative Humidity of the gas was simply the ratio of the lower pressure to the higher pressure, or at least very nearly. Since that time, a large number of humidity generators of similar type have been constructed both privately and commercially by a variety of institutions and companies. Many calibration and research laboratories throughout the world own and use humidity generators based on those original principles. While each of these generators exhibit differences in design, manufacturing, size, operation, automation, and the like, they all share a commonality in the basic principles which rely on the ability to generate an atmosphere of known humidity through the measurement and control of temperatures and pressures alone.

2. TWO-PRESSURE PRINCIPLE

In an ideal two-pressure system, a stream of gas at an elevated pressure is saturated with respect to the liquid or solid phase of water and then expanded isothermally to a lower pressure. Measurements of the pressure and temperature of the saturated gas stream, and in the test chamber after expansion, are all that is required to properly determine the resulting humidity content of the expanded gas stream.

A two-pressure generator is commonly used in the generation of a range of Relative Humidity values at fixed temperatures. Generally, the saturator and chamber share a common bath and are ideally in thermal equilibrium. In this case, the Relative Humidity produced is based primarily on the ratio of the measured saturated gas stream pressure (saturation pressure) to the measured chamber pressure using the simplified approximation

$$\%RH \approx \frac{P_c}{P_s} \cdot 100 \quad (1)$$

where P_c is the Chamber Pressure, absolute
 P_s is the Saturation Pressure, absolute

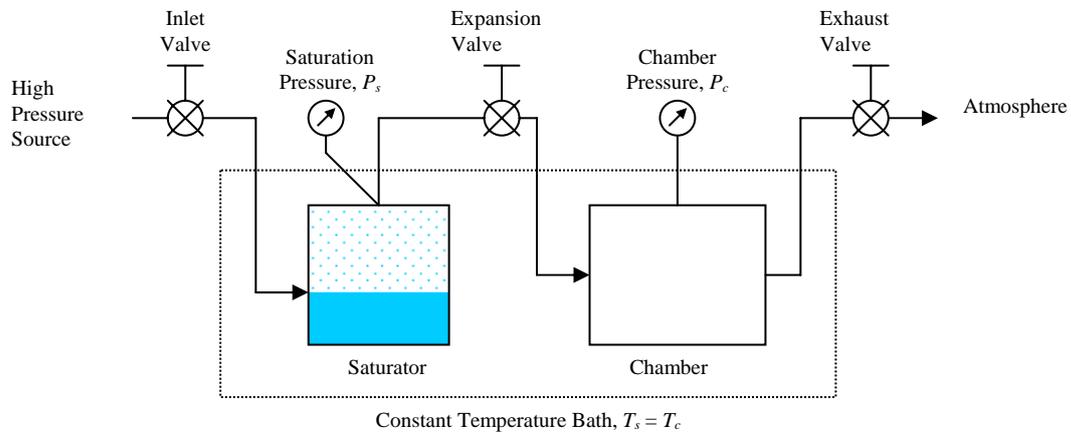


Figure 1: Simplified Schematic Diagram of the Two-Pressure Method where $T_s = T_c$

Accuracy of a two-pressure generator can be greatly enhanced by accurately measuring the temperatures of the saturator and chamber rather than relying on ideal assumptions of equality. Consider the system depicted in figure 2. In this system, a stream of gas at an elevated pressure is saturated with respect to the liquid or solid phase of water at a given saturation temperature. The gas stream is then expanded to a lower pressure and alternate temperature. To ensure full saturation, this system also uses a presaturator.

Measurements of the pressure and temperature of the saturated gas stream (P_s and T_s), and in the test chamber after expansion (P_c and T_c), are all that is required to properly determine the generated humidity at the test chamber.

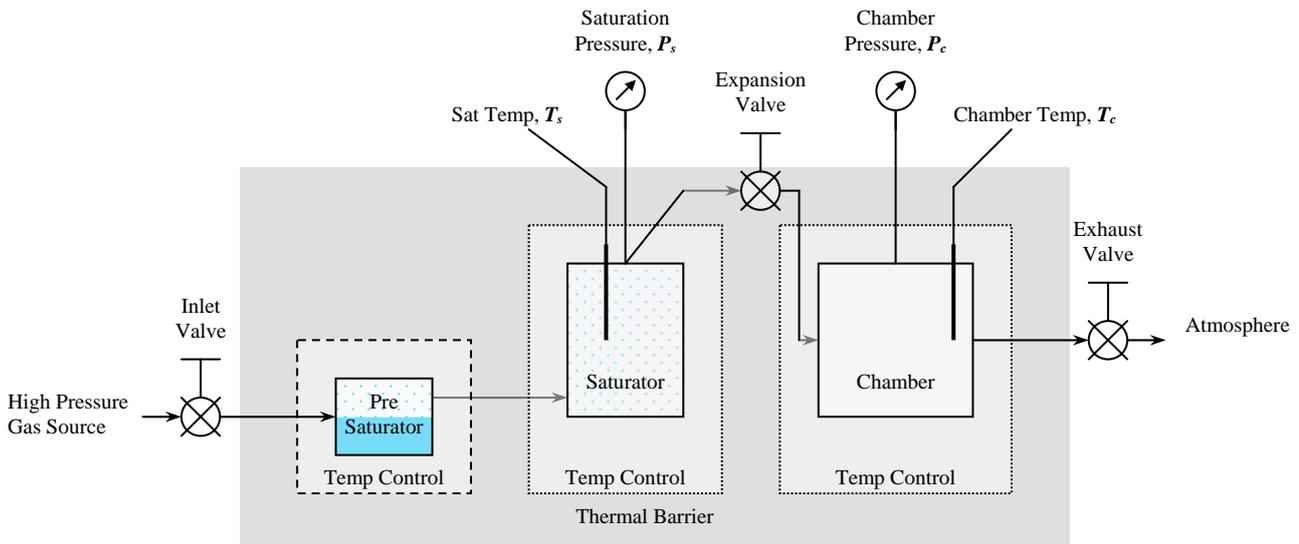


Figure 2: Schematic Diagram of the Two-Pressure Method

The diagram depicts a thermal barrier between the saturator and chamber temperature control sections. However, for simplicity a common temperature controlled medium is often used for controlling the saturator and chamber at a common temperature. While this simplifies the design

and control of such a system, independent measurements of the saturation and chamber temperatures should still be made and used in the determination of relative humidity. The four independent measurement parameters of saturation temperature, chamber temperature, saturation pressure, and chamber pressure, can then be used in the relative humidity formula

$$\%RH = \frac{P_c}{P_s} \cdot \frac{F_s}{F_c} \cdot \frac{E_s}{E_c} \cdot 100 \quad (2)$$

where P_c is the absolute chamber pressure,
 P_s is the absolute saturation pressure,
 E_c is the saturation vapor pressure computed at the chamber temperature,
 E_s is the saturation vapor pressure computed at the saturation temperature,
 F_c is the enhancement factor computed at the chamber pressure and temperature,
 F_s is the enhancement factor computed at the saturation pressure and temperature.

From the four parameters of temperature and pressure, all other humidity parameters may also be computed such as dew point, frost point, parts per million by volume and others. For purposes of this discussion, relative humidity will be the only parameter shown in mathematical detail but additional information may be found in the references.

3. SATURATOR DESIGNS

The saturator is a main component of humidity generators and is used to establish the saturation temperature and saturation pressure, two of the four key elements required for computation of most humidity parameters. The saturator must be designed properly to allow for adequate condensation, evaporation, and heat transfer. The designer must account for temperature and pressure range, humidity range, flow rates, and heat load, to name a few.

Regardless of the saturator design, each operates as a heat exchanger. The design should be adequate enough to heat or cool the gas stream to the desired outlet temperature, while fully saturating the gas stream with water vapor at that temperature condition. An accepted consequence of heat exchange is that the saturator inlet will be at a different temperature than the outlet. With most saturators, it is often very easy to define the direction of gas flow, since it usually flows from inlet to outlet in a fixed tube or vessel. These tubes or vessels are often placed in a fluid bath to control the temperature. However, proper control of the flow of this fluid path is often overlooked and is a common pitfall in the design of a humidity generator. Rather than simply placing the saturator in a temperature regulated stirred fluid bath, it is best to control the fluid flow path to be opposite that of the gas. So while the gas flows from saturator inlet to outlet, the thermally regulated fluid should flow in the reverse direction. This counter-flow design ensures the best chance for full heat exchange, and allows for better thermal control of the gas outlet temperature regardless of the temperature, pressures, or flow dynamics of the gas. Temperature of the fluid should be controlled at a point nearest the gas outlet.

Due to the many factors involved, a variety of saturator designs exist for different applications. The following are some basic saturator designs.

Submersed Coil Saturator

A submersed coil is generally constructed from a long continuous length of polished stainless steel tubing. It has been wound around a cylindrical object in order to form it into the shape of a coil. The coil is then prepared for use by flowing humidified gas through it (ambient atmospheric air for instance) while *slowly* (from several minutes to several hours per centimeter) submersing it into a liquid bath that is controlled at a temperature below freezing. Humidity from the flowing gas will

condense out as it cools to the temperature of the bath, thereby depositing a layer of frost on the inner walls of the coil. This slow incremental submersion process generally takes several hours to evenly frost the coil. The orientation of the coil is vertical so that incremental submersion causes the frost layer to evenly build upward along the spiral.

When adequately frosted, the humidified gas is replaced by dry gas flow. The coil is then submerged further to create a short barrier between the frost line and the top of the liquid bath to ensure that the frost is at bath temperature rather than at some warmer temperature due to heat piping near the bath surface. Finally, the bath is changed to the desired temperature (must be below freezing) to set the desired humidity level of the exhaust gas. The humidity is introduced to the gas from sublimation of the frost into water vapor. Depending on the generated humidity, and the dryness of the incoming gas, the coil may eventually dry out and require re-preparation. Preparation is always done at temperatures below freezing. The submersed coil saturator could also be operated in a condensation mode, but with significantly limited operating time due to eventual frost buildup and blockage of the coil.

Often chosen for their simplicity and ease of construction, submersed coils are used for generating gas with low flow rate and low humidity where the pressure-dew point is below 0°C in order to maintain the coil below freezing. Obviously, if the coil is allowed to warm above freezing, the frost melts and liquid water spirals to, and collects at, the bottom of the coil.

Adequate coil length must be used to ensure that the gas passing through the saturator has fully warmed or cooled to the coil temperature, and that adequate length exists for full saturation. But there is an upper limit to this length dictated by the flow rate and pressure drop across the coil. To prevent errors due to thermal gradients and pressure drops, the temperature and pressure measurements of the saturator should be made close to the coil outlet.

Dammed Coil Saturator

Variations of the normal submersed coil saturator exist whereby a series of special barriers are constructed within the tubing to dam liquid water at several locations throughout the tubing. The dams are high enough to pool or trap water, but low enough to prevent blockage and allow gas to flow over the top of each of them. This type of coil is prepared for use by injecting some liquid water at the top, draining any excess from the bottom, submersing the coil into the temperature controlled bath, allowing the dammed water to freeze, and flowing dry gas through it. Obviously, the orientation of the saturator is vertical.

As with the normal submersed coil saturator, this dammed coil saturator is generally used for generating gas with low humidity where the pressure-dew point is below 0°C. However, since the dammed coil can operate at temperatures above freezing, some limited applications are found in this region. The dammed coil saturator operates on the principle of either sublimation or evaporation, depending on the saturation temperature, as set by the temperature controlled bath. Also, depending on the generated humidity, and the dryness of the incoming gas, the coil may eventually dry out and require re-preparation. The water filling preparation is always done at temperatures above freezing.

Like the submersed coil saturator, temperature and pressure measurements of the saturator should be made close to the outlet.

Stacked Plate Saturator

The stacked plate saturator is constructed from a series of plates or dishes stacked vertically on top of each other. The plates are then sealed together on the perimeter. Each plate or dish is hollowed out or grooved in some fashion so as to hold liquid water, and each has strategically placed dams

and spill holes to allow for the drainage of excess water and for gas flow. The saturator is prepared for use by injecting water on the top plate and allowing it to percolate down to the remaining plates in the stack, then draining any excess. This filling process is performed while the saturator is above freezing. Plate temperature is then cooled to the desired operating temperature (generally below freezing), and dry gas flows from inlet to outlet across the plates to establish the desired humidity.

As with the normal submersed coil saturator, this stacked plate saturator is generally used for generating gas with low humidity where the pressure-dew point is below 0°C. However, since the plates can hold liquid water and operate at temperatures above freezing, some limited applications are found in this region. The stacked plate saturator operates on the principle of either sublimation or evaporation, depending on the saturation temperature, as set by the controlled plate temperature. Also, depending on the generated humidity, and the dryness of the incoming gas, the coil may eventually dry out and require re-preparation. Water filling preparation is always done at temperatures above freezing.

Bubbler Saturator

In a bubbler saturator, gas enters the bottom of a vessel and is allowed to bubble up through liquid water in order to achieve saturation. Bubblers can only operate at saturation temperatures above freezing and are generally used only for low flow rates applications. At higher flow rates, they are subject to the possibility of droplet carryover, aerosol, and/or spray, all of which tend to cause a higher than expected humidity output. This is due to micro-droplets of water being suspended in the gas stream and carried out with it. Mesh, filters, and screens are often used within bubblers to lessen these carryover possibilities. Lower flow rates and higher temperatures also tend to minimize the droplet carryover problem associated with bubbler-type saturators.

Condensation Saturator

Unlike the coil and plate type saturators which present a large surface of frost or water for purposes of adequate evaporation, the condensation saturator must act more as a heat exchanger to cool the gas and force it to condensation. For this reason, the condensation saturator is more adequately described as a heat exchanger. It must be able to take gas, at the highest designed flow rate and temperature for the system, and cool it to the desired saturation temperature in one pass. Tube-in-shell, or tube-bundled, heat exchangers are often used as condensation saturators.

A condensation saturator requires a presaturator capable of taking dry gas and delivering warm, pre-humidified gas. The gas from the presaturator is said to be *super-saturated* with respect to the cooler temperature of the condensation saturator. The condensation saturator, in reality a heat exchanger, cools the gas down to a desired level. Any water vapor in excess of that allowed for full saturation at that temperature condenses out.

While orientation of condensation saturators is not of concern, gas exit should generally be from the top or sides. In addition, some method must exist for removing the condensate from the bottom of the saturator to prevent it from filling. One simple but effective method is to mount the saturator physically above the presaturator so that gravity can allow the condensate to run back into the presaturator. Assuming the original source gas entering the presaturator is drier than the final generated gas from the system, there is always a net loss of system water so there is no danger of overfilling the presaturator from the saturator condensate. However, since there is a net loss of water, some method must also exist for replenishing water in the presaturator.

In contrast to the coil and plate type saturators previously mentioned, condensation saturators are normally used for generating mid to high humidity values where the saturator may remain at a temperature above freezing. They are also used for much higher flow rate applications, often as much as one or two orders of magnitude higher than coil, plate, and other evaporation mode

saturators. Occasionally, condensation-type saturators are operated at temperatures below freezing, but suffer at those sub-freezing temperatures from limited run time due to buildup of frost and eventual blockage of the gas flow.

Presaturator

Use of a presaturator causes a gas to become super-saturated with respect to the desired output (or saturation) temperature by warming the gas to an elevated temperature and raising its humidity level toward saturation at that temperature. While full saturation at the elevated temperature is rarely achieved or even desired, the water vapor content achieved in the gas at that elevated temperature must be higher than what is required for full saturation at the temperature of the saturator (which is always cooler than the presaturator). When the gas is presaturated with water vapor at a warm temperature, then subsequently cooled to the saturator temperature, condensation of the excess (in other words full saturation at the saturation temperature) will occur. Presaturator designs need not be elaborate, but must incorporate some method for elevating temperature and humidity. A presaturator can be as simple as a heated vessel, partially filled with water, through which the gas flows. A more elaborate presaturator may incorporate pumping and stirring methods designed to enhance the evaporation of water into the gas.

Allowing the operator to vary the temperature, pumping, or stirring of a presaturator can enhance the ability to test the adequacy or efficiency of condensation saturator designs. For instance, the presaturator temperature may be lowered until a drop in the generated humidity output is noticed. Conversely, presaturator temperature may be raised until an increase in humidity output is noticed. Another approach is to raise presaturator temperature until the measured temperature of the saturator begins to increase. This establishes the threshold temperature whereby further increase overdrives the heat exchanging capability of the saturator, attempting to work it beyond its capability. The lower and upper presaturator temperatures determined from these tests establish the working temperature range of the presaturator for a specific saturator design operating at a specific set of temperature and flow conditions. Similar tests may be conducted by varying stirring and/or pumping of the presaturator if available within the particular presaturator design.

4. TEMPERATURE AND PRESSURE MEASUREMENTS

Saturation Temperature

The saturation temperature measurement should be taken at or near the saturator gas outlet. However, heat piping, stem conduction, and self-heating are likely sources of temperature measurement errors in systems operated at low flow rates or operated at temperatures significantly above or below ambient temperature. In these cases, the very act of directly measuring the saturated gas may induce measurement errors. In these cases, it is often acceptable to measure the temperature of the controlled medium surrounding the saturator as close as possible to the saturator gas outlet. When the system has reached thermal stability and operational equilibrium, the temperature of the controlled medium is assumed to be an adequate representation of the actual gas temperature within the saturator. Regardless of the placement of the thermometer, care should still be taken to eliminate to the extent possible all measurement-induced errors.

Chamber Temperature

The chamber temperature measurement should be taken at a point in very close physical proximity to any humidity measurement device under test. In the case of multiple humidity devices under test, choose a central location representative of the group. Care must be taken to avoid such measurement errors as heat piping, stem effect, and self-heating. If the chamber is sufficiently large, placing more of the cable inside the chamber can minimize thermometer stem effect. This isolates the thermometer element, by way of physical cable length, from the temperature outside the chamber. To lessen the effect of heat piping, use smaller, less bulky cables when possible both

for the thermometer and any humidity devices under test. This will lessen the thermal transfer of heat through cables between inside and outside the chamber.

While often over-looked, relative humidity is highly temperature dependent. When generating relative humidity, thermal gradients in the chamber result directly in humidity gradients. Care should therefore be taken to ensure uniformity of the chamber temperature. Another approach is to place separate thermometers at various locations throughout the chamber, then compute relative humidity at each of these locations. Placing thermometers in very close proximity to any humidity sensors under test is another alternative whereby the local humidity in the micro-environment surrounding the sensor can be computed. Fans can also be used to stir the air within the chamber in an attempt to provide for better thermal homogeneity, but at the expense of added heat due to the fan motor. Operating the fan at lower speed with lower power is often preferable to reduce the effects of fan induced heat. A balance must be met between fan speed and induced heat.

When generating relative humidity, the absolute accuracy of temperature measurements is generally of less concern than the relative accuracy between the saturator and chamber temperature measurements. In other words, both the saturator and chamber temperature measurements may be in error as compared to an absolute reference, but if they match each other well, then the relative humidity will still be accurately generated since temperature measurements are used ratiometrically in the relative humidity formula (see equation 2). If generating any other parameter, such as dew point, then absolute accuracy is required in temperature measurement.

Saturation Pressure

Measure saturation pressure as close as possible to the saturator gas outlet. To avoid significant thermal drift, mount the transducer in an area thermally isolated from the saturator, and connect the transducer via tubing. Avoid kinks and crimps in the tubing. Ensure all connections are gas tight. Although inevitable, avoid dead volumes to the best extent possible, especially in low flow, low humidity applications. In the computation of humidity parameters, all pressure measurements must be absolute rather than gauge.

Due to the wide dynamic pressure range of most saturators, more than one transducer may be required to adequately cover the range while still maintaining accuracy at the lower pressures where accuracy is of most importance. When selecting a series of transducers to cover a wide dynamic range, a series of uncertainty calculations should be performed to determine the applicable ranges for each transducer. A method is also required of pneumatically switching out the lower range transducers during those times that the saturator pressure exceeds their operating range. The switch system must ensure that the proper range transducer is both pneumatically and electrically activated.

Chamber Pressure

While not a requirement of the humidity generation principles, most chambers operate at or very near ambient pressure. Select a transducer appropriate for the chamber's operating pressure range. For chambers of wide dynamic temperature, thermally isolate the transducer from the chamber, making the pneumatic connection via tubing. Avoid kinks and crimps in the tubing and ensure all connections are gas tight. Although inevitable, avoid dead volumes to the best extent possible in low flow, low humidity applications. In the computation of humidity parameters, all pressure measurements must be absolute rather than gauge.

When generating high relative humidity, most designs result in nearly equal pressure in the saturator and chamber. If the transducers used for the saturator and chamber pressure measurements have unequal zero drift, a significant error in generated humidity will result. One method for avoiding this problem is to time-share a transducer between the chamber and the saturator. Even if the time-shared transducer sees drift, the drift will be of the same sign and will

result in nearly equal offsets for the chamber and saturator measurements resulting in a smaller error in generated humidity output.

5. VERIFICATION

Regardless of the design, the output of a humidity generator should be verified to ensure that the theory and implementation agree within acceptable limits. To accomplish this, humidity measurements should be taken with a suitable humidity measurement device, most commonly a chilled mirror hygrometer. The measurements may simply provide a method for detecting probable system malfunction, or in the case of a highly accurate measuring instrument they may be used for direct verification of the generated humidity. Two important tests, saturator efficiency and humidity verification, should be considered.

Saturator Efficiency

A saturator should be tested in order to establish its operating limits, and to the best extent possible, its efficiency. Ideally, a saturator should be 100% efficient under all operating conditions, however many may not be. Saturator verification involves testing the saturator at a variety of conditions. The conditions most useful for testing are often those expected to provide the saturator's worst operational output, such as those of higher temperature and higher flow rates. Both conditions can cause saturator inefficiencies due to failure of the saturator to adequately saturate the gas stream with water vapor. Both conditions often result in lower than expected water vapor content in the gas.

A chilled mirror hygrometer is often used to test saturator efficiency by direct measurements of the saturator output. This requires a highly sensitive, repeatable, and accurate instrument. To determine saturator efficiency requires the measurement of saturation temperature and dew (or frost) point. From these two measurements a ratio of the ideal vapor pressure to the measured vapor pressure establishes the saturator efficiency.

$$\text{Saturator Efficiency} = e_{ideal} / e_{actual} \quad (3)$$

Where e_{ideal} = saturation vapor pressure computed at the saturation temperature, and
 e_{actual} = saturation vapor pressure at the dew point temperature.

If the saturator efficiency is not 100%, then consideration should be given to redesign of the saturator to better match the operating parameters. When redesign is not possible, the relative humidity output of the generator should be corrected for the inefficiency with

$$\text{Corrected RH} = \text{Estimated RH} * \text{Saturator Efficiency} \quad (4)$$

Where *Estimated RH* is computed from equation 2, and
Saturator Efficiency is computed from equation 3.

Verification of Humidity Output

After verification of saturator efficiency, and determining it adequate for the design, the output of the system should be tested. As with saturator verification, the chilled mirror hygrometer is often the instrument of choice. Highly sensitive and accurate instruments simplify this testing. However, for the case of a two-pressure two-temperature generator, absolute accuracy of the hygrometer is less important than sensitivity and repeatability. Since the generator can be used to generate the same theoretical dew or frost point temperatures under a variety of temperature-pressure combinations, a single fixed dew or frost point value can be chosen as the test value. By generating the test value with various saturation temperature and saturation pressure combinations, the relative difference in observed hygrometer readings can be noted and used to verify the output

of the system. Concerned with only the relative differences, the absolute hygrometer accuracy is of little importance provided the hygrometer maintains a high degree of repeatability.

When using chilled mirror hygrometers, it is very important to understand the dew point vs. frost point relationship for values below 0°C. When operating below 0°C, the water vapor may condense as liquid (termed super-cooled dew), or it may condense in the form of ice (also called frost). While the vapor content of the gas is the same, the temperature associated with liquid condensation will differ from that of ice condensation. Whether the gas condenses as dew or frost is not a function of the generating method, saturator temperature, saturator type, saturator state (liquid or ice) or other generator factors. It is a function of the chilled mirror hygrometer.

Consider the output of a humidity generator which has been set for -10°C dew point. Note that it is of no importance how this value is generated, or the conditions (liquid or ice) within the saturator that produce this result. For this explanation, simply accept that the output of the generator is -10°C dew point. Using an accurate chilled mirror to measure this generated humidity will likely result in one of two different measurement readings; -10°C if the water vapor condenses on the mirror as super-cooled dew, or -8.9°C if the water vapor condenses on the mirror in the form of frost. Both indicated results are correct, provided that the state of the condensate on the mirror is known. It can not be assumed that simply because the reading is lower than 0°C that the result is frost point. In fact most chilled mirrors will first exhibit dew point and hold in that meta-stable state for some time, finally freezing and then indicating a temperature of frost point. The amount of time required for this transition from dew to frost is often long and unpredictable. It is based on a variety of factors such as the cleanliness of the mirror, the temperature of the mirror, and the stability of the gas flow. Once a condensate layer has transitioned to frost, it will not revert back to dew provided that (1) the mirror remains below 0°C (in other words the frost does not melt), and (2) the mirror does not lose its condensate layer.

This dew point vs. frost point difference is most often observed in the range between 0 and -30°C. The difference between dew and frost point measurements in this range, for measurements of the same gas content, is approximately 1 degree in every ten. Near 0°C, there is no perceptible difference. For dew point of -30°C, the equivalent frost point temperature would be approximately 3°C warmer, or approximately -27°C frost point. When operating below mirror temperatures of -40°C, the probability of maintaining super-cooled dew on the mirror falls off dramatically, and all chilled mirrors will likely exhibit frost point rather than dew point.

When using a chilled mirror to verify the humidity output from a generator, it is important to know whether the mirror is controlling on super-cooled dew or on frost. The uncertainty associated with any lack of this knowledge will likely dominate in any uncertainty analysis. However, methods for proper dew/frost determination can be employed, such as the use of an endoscope for visual inspection of the condensate. With some experience viewing difference dew and frost layers, an operator can often visually determine the difference between dew and frost. An alternate, and perhaps better method, is the use of a chilled mirror containing the ability to force the condensate into frost and properly distinguish frost from super-cooled dew. By correctly distinguishing super-cooled dew from frost, the large uncertainty associated with this measurement can be eliminated.

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