Solving Humidity Calibration Challenges in Today's Metrology Lab

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Introduction/Abstract

Two-pressure humidity calibration technology has long been the recognized standard for on site instrumentation calibration, test and verification. The goal of this paper is to provide a tool to help you as a metrologist take advantage of the benefits of this technology so you will be able to apply the information to your daily laboratory applications. This paper will cover the operation and benefits of modern two-pressure humidity calibration systems, explain how you calibrate them, provide some sample applications and a brief history of the technology.

The Importance of Reliable Humidity Calibration in Your Lab

Whatever your industry, your end product is only as good as the calibration it has received. Therefore, the equipment used to calibrate or test the limits of your end product must be first, a reliable, proven technology and, second, be presented in equipment that is easy to use and also easy to validate for optimum operation parameters; preferably at your laboratory, in-house.

Whether you are calibrating transducers, tools for silicon wafer production, controlling comfort levels in HVAC systems or are involved with the tight humidity management required in manufacturing moisture sensitive products such as film, semiconductors, and pharmaceuticals, you are stepping up your demand for increased reliability and accuracy in humidity measurements. Today, calibration systems are required to both obtain and maintain a 4:1 accuracy ratio. To accomplish this, humidity and dew point hygrometers must be calibrated against a source of humidity at a stable test temperature.

The most accurate and reliable method of continuous humidity generation in use today for the range of ~5-98% RH is based on the "two-pressure" principle originally developed by the National Institute of Standards and Technology (NIST). The two-pressure principle is used in the most accurate on-site calibration and verification systems. These are mobile and self-contained, with an integral humidity generator that is capable of simulating a wide range of temperature/humidity values with sufficient accuracy and consistency to maintain strict 4:1 calibration ratios. In a 10-year track record, this has been the only system of its kind that can meet tight tolerance requirements. It is still the primary standard of choice for humidity calibration.

Understanding How the Two-Pressure RH Generator Operates

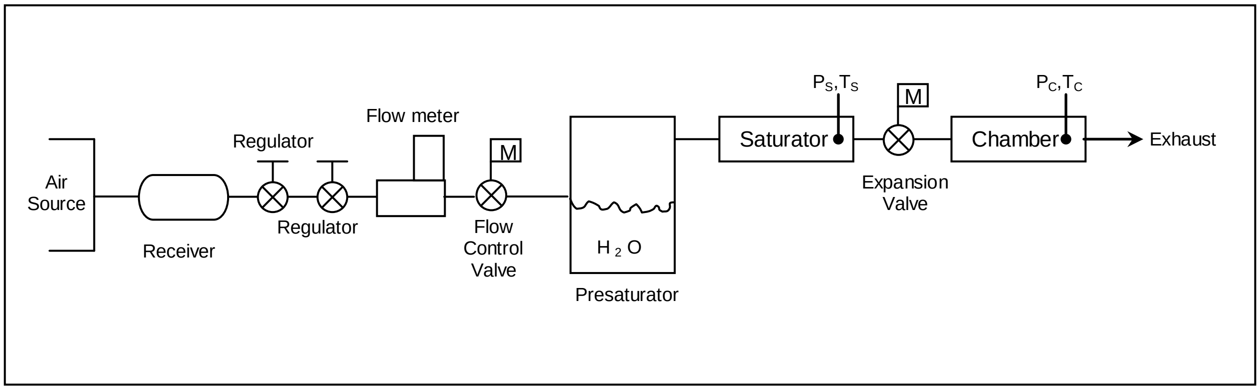

Figure 1. Operational flow schematic.

The best way to describe operation is by dissecting the actual operational technology of an established system. We will use the Model 2500 two-pressure humidity generator for this purpose. Model 2500 is a portable, self-contained, two-pressure humidity generator that uses compressed air of up to 175 psia (1207) kPa) provided by either a portable oil-free air compressor or other equal source and directed to a receiver. The air then passes through dual regulators, achieving regulated pressure of ~ 150 psia (1034 kPa) and is directed to a flow control valve. Although the humidity does not depend on flow rate, the flow control valve is adjusted to set an airflow rate of 2-20 slpm through the system. The flow rate is monitored by a flowmeter installed directly upstream of the flow control valve. Then the gas flows to a presaturator.

The presaturator is a vertical cylinder partially filled with water that is maintained at a temperature ~ 10-20 °C above the desired final saturation temperature. Air entering the presaturator first flows through a coil of tubing immersed in the water, a configuration that forms a heat exchanger. As the air passes through the immersed tubing, it is warmed to “at or near” the presaturator temperature. Air exiting the tubing is deflected downward onto the water surface in a manner that causes circular airflow within the presaturator. While passing through the presaturator, the gas continues to warm to the presaturator temperature and becomes saturated with water vapor to nearly 100% RH.

Next, the gas flows to the saturator, which is a fluid-encapsulated heat exchanger maintained at the desired final saturation temperature. As the nearly 100% RH gas travels through the saturator, it begins to cool, forcing it to the dew point or 100% saturation condition. The gas continues to cool to the desired saturation temperature, causing moisture in excess of 100% to condense out. Forcing condensation ensures 100% humidified gas. The saturation pressure P_S and the saturation temperature T_S of the gas are measured at the point of final saturation before the gas stream exits the saturator.

The gas then enters the expansion valve, where it is expanded to a lower pressure, which is the test chamber pressure P_C. Because adiabatically expanding gas naturally cools, the valve is heated to keep the gas above dew point while it expands to the lower pressure. If the gas or the valve were allowed to cool to or below the dew point, condensation could occur at the valve and alter the humidity content of the gas. The cooling effects of expansion, while mostly counteracted by the heated valve, are fully compensated by flowing the gas through a small post-expansion heat exchanger. This allows it to reestablish thermal equilibrium with the fluid surrounding the chamber and saturator before it enters the test chamber. The final pressure P_C and temperature T_C of the gas are measured within the test chamber. The test chamber exhausts to atmospheric or ambient pressure and so is very near ambient pressure.

A computer/controller embedded in the system controls the entire humidity generation process: temperatures, pressures, and system flow rate. It also handles keypad input, parameter measurements and calculations, data display, and external I/Os to link to peripherals such as additional computers or printers.

Temperature Control: Every humidity generating process requires precise temperature control (setpoint) and good temperature stability. These are ensured by digital computer control of the temperature of a circulating water/glycol mix that jackets the saturator and test chamber areas of the generator. The saturation and chamber temperatures are governed by the temperature of this medium. The computer will keep this at any value from 0 to 70° through the use of PID (proportional – integral – derivative) algorithms.

The PID algorithm compares the measured temperature to the desired setpoint temperature to calculate the temperature difference (proportional); the current rate at which the temperature is changing (derivative); and the accumulation of the temperature difference over time (integral). Each calculation is effectively multiplied by an associated weighing factor, and the three are then added together to provide a numerical value. This value, termed the PID output, represents the percentage of the total available heating or cooling capacity required at a given time. The value is recalculated approximately once each second and is used to time-proportion heating and cooling devices. In short, the PID output determines how long to apply power to a specific heating element or how long to open a refrigeration or coolant solenoid during each one – second interval.

The fluid medium is heated by time-proportioning an immersion heater in the fluid circulation path. Cooling, while also time-proportioned, is accomplished by injecting a high-pressure liquid refrigerant (R-134) from a closed compressor system into a heat-exchanging evaporator in the fluid circulation path. Using PID algorithms for temperature control allows the fluid temperature to be maintained at the desired saturation temperature with a stability to within ~ 0.02 °C over the operating range.

The presaturator temperature is similarly controlled by time proportioning. Heating is done by applying power to an immersion heater and is bucked merely by the ambient temperature of the incoming air.

Pressure Control: A computer controlled electromechanical valve assembly controls the pressure control of the saturator. Saturator pressure is measured at ~1 sps and is used as data in PID algorithms similar to those employed in temperature control. The algorithms determine the required valve position.

Conventional two-pressure generators incorporate three separate pressure transducers: one for chamber pressure, one for lower saturator pressures and one for higher saturator pressures. The problem with this approach is that at low saturator pressures, a dual-drift effect (offset drift between the chamber and low-pressure saturator transducer) can cause significant errors in the calculated RH.

The generator discussed here solves this problem by using only two transducers: one low-range for chamber and lower saturator pressures, and one high-range transducer for higher saturator pressures. The low- range transducer is time-shared between the chamber and the saturator when it is operating at lower pressures. Time-sharing of the low-range transducer eliminates the dual drift often seen when using separate chamber and low-range saturator pressure transducers.

Validating Your Calibration System

It goes without saying that the system you use to calibrate and test your product must, itself, be in prime parameters. The easier it is to validate for optimum operation and calibrate to strict specifications, if necessary, the more value is added to the equipment. If a system must be sent outside for recalibration or a specialist must be brought in, it adds to your cost of ownership.

The ability to validate and recalibrate laboratory equipment in-house should be high on any metrologist’s priority list. It is not cost effective to have equipment in your lab that you can’t calibrate yourself.

Simple Calibration

Since proper calibration of the temperature and pressure transducers ultimately determines the accuracy of a two-pressure humidity generator, a good portable system employs an integral programmatic calibration scheme. Rather than removing transducers from the system and sending them to a laboratory for calibration, you just take the entire system to your lab or bring the appropriate pressure and temperature standards to the system. You calibrate the transducers while they’re electrically connected to the humidity generator. This “in the system, as a system” approach helps eliminate systemic errors that might be induced by other calibration methods. Because all calibration is performed mathematically by the computer, manual adjustments are not needed.

Calibration is performed on each transducer by the computer solution of the coefficients ZERO, SPAN, and LIN to this simple quadratic formula:

$$Y = LIN \bullet X^2 + SPAN \bullet X + ZERO \quad (2)$$

Where:

X = raw count (or output) of the A/D converter while measuring the transducer.

Y = desired value (the standard or reference transducer reading) for the transducer being measured.

The coefficients ZERO, SPAN and LIN are found by applying three separate, distinct, and stable references to each transducer and then solving the resulting mathematical system of three equations with three unknowns. Since all the measurements and calculations are performed automatically by the embedded computer, you only need to provide the three known stable references: one near the low end, one near the center, and one near the upper end of each transducer's intended range.

For a low-end temperature calibration point, you take the temperature bath to a low point, ensure stability, and then enter the value indicated by a standard or reference thermometer. Repeat this procedure at two additional points: near the middle and upper ends of the temperature range.

When the three reference points have been applied, the new coefficients for each probe are displayed. The coefficients for each transducer are stored in the system's nonvolatile memory until the next calibration is performed.

You should run intercomparison validations on a regular basis. These tests must compare your equipment against a chilled-mirror hygrometer, psychrometer or other known consistent humidity-measuring device. Use a variety of humidity values and temperatures for this validation, and keep current control charts on all the results to ensure they are within the estimated uncertainty. Make sure this includes both normal trends and abnormalities because this is the most accurate record you will have to indicate if temperature probes or pressure transducers start to drift from their required calibration. Drifts will also warn you of other operational faults, including water or heating problems in the presaturator or saturator. Water contamination, leaks in the gas path and numerous other issues will also show up clearly if you have a basic tracking schedule established to easily pinpoint whenever points or out-of-spec drifts occur. This should be part of your overall preventative statistical process control (SPC) to catch abnormalities before they can cause problems.

Field Trials and Test Data

A relative humidity uncertainty analysis[2] was conducted on the Model 2500 portable, self-contained, two-pressure humidity generator used for data in this presentation, following NIST Guideline 1297. The relative humidity in a two-pressure humidity generator of this type is determined from the measurements of temperature and pressure only using the following formula:

$RH = P_c / P_s * E_s / E_c * F_s / F_c * 100$ where P_c = Chamber Pressure,

P_s = Saturation Pressure,

E_s = Saturation Vapor Pressure at Saturation Temperature,

E_c = Saturation Vapor Pressure at Chamber Temperature,

F_s = Enhancement Factor at Saturation Temperature and Pressure,

F_c = Enhancement Factor at Chamber Temperature and Pressure,

100 = nominal saturator efficiency.

The study was concerned with analysis of the above ratios separately and then combined, within four specific categories of uncertainty: contribution from the pressure ratio term P_c / P_s contribution from the vapor pressure ratio term E_s / E_c; contribution from the enhancement factor ratio F_s / F_c, and contribution from saturator efficiency.

This analysis was conducted to validate the accuracy of performance using temperature and pressure uncertainty calculations. These uncertainty calculations of the Model 2500 two-pressure humidity generator served to establish that the system is within the manufacturer's stated specification and that traceability can be established with NIST. Full analytical details of the study are available on the Thunder Scientific Corporation website.[3]

Application Examples

Portable two-pressure humidity generation calibration equipment is in heavy use in pharmaceutical, aerospace and semiconductor applications. It's also the number one system used by sensor manufacturers. US Air Force, US Army and US Navy metrology or "PMEL" laboratories use this type of equipment for humidity calibration standards. The technology is also found in regular use in pharmaceutical production, semiconductor clean room monitoring sensors, medical laboratories and in HVAC environmental controls. The range of applications is extremely wide. The following are only a few examples:

<u>Chart Recorders:</u> A test chamber can typically accommodate two standard size hygrothermographs. Temperature/humidity data can be run at virtually any points desired and for any length of time. Charts can then be compared with the printer output for analysis and adjustment. Once adjusted, either the same points or others may be run again for verification. Onsite calibration eliminates rough handling and exposure of the recorder to undesirable temperature/humidity extremes. In addition, because temperature is variable (even while maintaining constant RH), temperature sensitivity is easily determined.

<u>Chilled Mirror Hygrometers:</u> A humidity computer can be used to determine either the saturation pressure or RH necessary to generate a specific dew or frost point. First, the generator is run to allow most of the gas to exhaust to ambient through the chamber vent. A small sample is drawn through the side port of the chamber, next through the chilled mirror head, and then through an adjustable valve or flowmeter. Because the chamber naturally operates at a very small positive pressure, flow rates of ~ 1 slpm through the chilled mirror head are easily obtainable. Flow rate through the head may also be adjusted by partially restricting the chamber exhaust.

The entire head can also placed in the chamber with the head exhausting to ambient. The slight positive chamber pressure forces a small flow of gas through the head. Again, a flowmeter should be used downstream, with flow adjustments made either with a valve or by partial restriction of normal chamber exhaust.

<u>Environmental Testing:</u> A portable two-pressure humidity calibrator can serve as a test bed for evaluation and R&D of humidity and/or temperature-sensitive products such as; plastics, composites, film, tobacco, blood gas analysis, pharmaceuticals, soil hydrology, consumables, electronics, and optics. Depending on the temperature and humidity being generated, the system may operate continuously from hours to months; the only limiting factor is typically the 1-gallon capacity of the internal distilled water reservoir used by the presaturator to humidify the air stream. With continuous generation of a nominal 50% RH at 21 °C, the reservoir will last about two weeks between refills. When generating dry cold gas, e.g., 10% RH at 0 °C, continuous operation is possible for more than nine months.

Portable two-pressure humidity generation calibration equipment is also a valuable tool in humidity sensor research and development, hygrometer calibration, certification, and humidity sensor original calibration certification. This technology is also critical in special long-term environmental exposure tests for weather related calibration of atmospheric and land-based humidity sensor instrument packages and for large volume humidity sensor calibration production.

History of Two-Pressure Humidity Calibration

Older methods of on-site verification were accomplished by either using a portable transfer instrument or conducting full laboratory calibration. But, using a portable transfer instrument that is first calibrated in the laboratory and then moved to the site for comparison only provides a best ratio of comparison around 1:1. Although more accurate, full laboratory calibration using humidity-generating equipment requires removing the instrument to be calibrated from its installation, transporting it to the lab and then replacing it after it is fully calibrated, which can cause a wide range of measurement errors.

Over the years, the National Bureau of Standards (now the National Institute of Standards and Technology – NIST) worked to solve these problems by developing a two-pressure humidity calibration technology that would eventually become the commercial device seen today in most labs worldwide. The origin of the commercial device in use today was a device developed in 1948 by E.R. Weaver and R. Riley at the National Bureau of Standards that utilized pressure rather than water vapor for the generation and control of humidity.

The Riley-Weaver two-pressure device utilized air or some other gas saturated with water vapor at a high pressure and then expanded to a lower pressure while kept at a constant temperature. The resulting relative humidity of the gas was the ratio of the lower pressure to the higher pressure.

That method was improved upon by A. Wexler and R.D. Daniels, also at NBS, in 1951 with the addition of temperature control. Using temperature control enabled Wexler and Daniels to saturate a gas with water vapor at a given temperature and then raise the temperature to a higher value, allowing the measurement of temperature and pressure to be used to determine the relative humidity.

The combined two-pressure, two-temperature humidity generators in commercial production today allow independent control of temperature and pressure. This device has been identified by NCSL International as an intrinsic/derived standard since the value of relative humidity is a mathematical relationship based on pressure and temperature.

The basic principle for the NIST Mark 2 humidity generator involves saturating a continuous stream of air or some other gas with water vapor at a given pressure and temperature. The saturated gas then flows through an expansion valve where it is expanded to a lower pressure. The resulting RH of the gas is then determined by the formula:

$$\%RH = \frac{f_W(P_S,T_S)}{f_W(P_C,T_C)} \bullet \frac{e_W(T_S)}{e_W(T_C)} \bullet \frac{P_C}{P_S} \bullet 100 \quad (1)$$

where:
f_W = enhancement factor
e_W = saturation vapor pressure
P_S = saturation pressure
P_C = chamber pressure
T_S = saturation temperature
T_C = chamber temperature

The RH generated by the two-pressure principle only depends on the pressure and temperature of saturation and on the temperature after expansion. When these factors are measured and controlled it permits precise control of the generated humidity. Also, because the humidity generated is based solely on the fundamental principles of temperature and pressure, no humidity sensors are needed to measure it.

NCSL International has published a Recommended Practice for Intrinsic/Derived Standards (RISP-5) on the two-pressure, two-temperature humidity generator.[1]

Conclusion
Increasingly stringent testing and calibration will be needed to meet the requirements of new technologies being developed for a wide range of instrumentation and devices — some yet unknown. As a metrologist, you must always stay one step ahead of these requirements for your specific industry. Today, you can be assured that at least in the area of humidity calibration and testing, the technology will allow you to keep pace with your accelerating industry requirements.

Two-pressure humidity generation technology is proven and traceable to NIST standards and the portable equipment integrating the technology has been designed to meet the strict requirements of all laboratory humidity calibration applications. Better yet, this equipment can be easily validated and recalibrated in your lab without calling in your original outside vendor. When calibration is done, you can be satisfied that your system will meet our toughest specification, just as it did when it was first delivered it to your lab.

This translates into ultimate reliability: the capability of modern portable two-pressure humidity generator/calibration systems to provide the highest standard for all laboratory calibrations on a continuing basis.

1 - RISP-5 is available from NCSL International, 1800 30th Street, Suite 305B, Boulder, CO 80301-1026, tel 303-440-3339, fax 303-440-3384. A full list of NCSLI Recommended Practices and other metrology training information is available at www.ncslinternational.org.

2 - Relative Humidity Uncertainty analysis of the Thunder Scientific Model 2500 Two-Pressure Humidity Generator, by Bob Hardy, Thunder Scientific Corporation, Albuquerque, NM, USA.

3 – Thunder Scientific Corporation website www.thunderscientific.com