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Solving Humidity Calibration Challenges in Today's Metrology Lab

As a metrologist, you know you need to calibrate your humidity sensing equipment. But do you know how easy this exercise can be?

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Because your end product is only as good as the calibration step, your calibration equipment must be based on reliable and proven technology. It should also be easy to use and validate—preferably in your own lab. These guidelines apply to transducer calibration, HVAC system control, and the tight humidity management required for making moisture-sensitive products such as film, semiconductors, and pharmaceuticals.

Figure 1. The Model 2500, a self-contained mobile two-pressure humidity-standard generator, features an air compressor accessory.

Two-Pressure Humidity Calibration

Today's calibration systems must both obtain and maintain a 4:1 accuracy ratio. To do 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 for the ~5%–98% relative humidity (RH) range is based on a two-pressure principle originally developed by the National Institute of Standards and Technology (NIST). The most accurate on-site calibration and verification systems are mobile and self-contained, and incorporate a humidity generator that can simulate a wide range of temperature/humidity values accurately and consistently enough to achieve that 4:1 ratio.

The Two-Pressure RH Generator

The best way to explain the operating principle of a two-pressure RH generator is to examine an established example. The Model 2500 (see Figure 1) uses compressed air of up to 175 psia (1207 kPa) provided by either a portable oil-free air compressor or other source and directed to a receiver (see Figure 2, page 40). After passing through dual regulators that produce a regulated pressure of ~150 psia (1034 kPa), the gas travels through a flowmeter and into a flow control valve. Although humidity is not affected by flow rate, the valve is set to maintain 2–20 slpm through the system.

The gas then flows to a presaturator, a vertical cylinder partially filled with water maintained at ~10°C–20°C above the desired final saturation temperature. Gas entering the presaturator flows through a coil of tubing immersed in the water, a configuration that forms a heat exchanger. As it passes through the tubing, the gas is ▶

warmed to "at or near" the presaturator temperature. Gas exiting the tubing is deflected downward onto the water surface in a manner that causes circular airflow within 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, 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. This step ensures 100% humidity. The saturation pressure, P_S, and the saturation temperature, T_S, of the air are measured at the point of final saturation before the air stream exits the saturator.

The gas then enters the expansion valve, which causes it to fall to the test chamber pressure, P_C. Because adiabatically expanding gas naturally cools, the valve is heated to keep the gas above dew point. 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 in the test chamber. This 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.

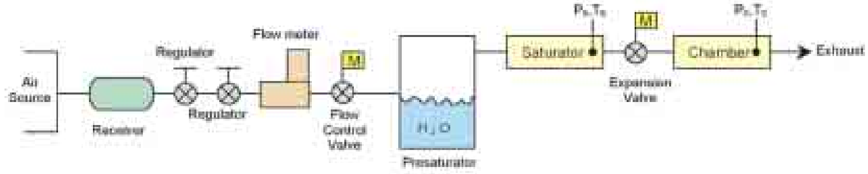


Figure 2. Two-pressure humidity generation entails saturating air or nitrogen with water vapor at a known temperature and pressure. The saturated high-pressure air flows from the saturator and through a pressure-reducing valve, where the gas is isothermally reduced to test pressure at test temperature. Humidity generation does not depend on measuring the amount of water vapor in the air, but instead is dependent only on the measurements of temperature and pressure.

Temperature Control

Every humidity-generating process requires precise temperature control (set point) and good temperature stability. These are ensured by using a digital computer to control 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°C to 70°C by means of PID (proportional-integral-derivative) algorithms.

The PID algorithm compares the measured temperature to the desired set-point 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 weighting 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 1 s 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

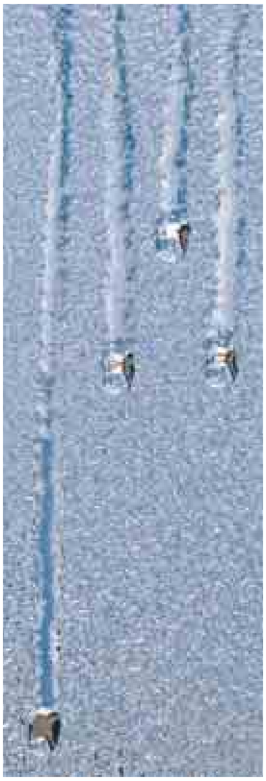
A computer-controlled electromechanical valve assembly manages the saturator pressure. This pressure is measured at ~1 sps and is used as data in PID algorithms similar to those used in temperature control. The algorithms determine the required valve position.

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 for higher saturator pressures. The low-range transducer is time-shared between the chamber and the saturator when it is operating at lower pressures. Such time-sharing 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 current calibration. The easier it is to validate for optimum operation and, if necessary, to calibrate to strict specifications, the more value is added to the equipment. If a system must be sent outside for recalibration or a specialist must be brought in, your cost of ownership goes up. 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 cannot 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 relies on an integral programmatic calibration scheme. Rather than removing transducers from the system and taking them away 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 are electrically connected to the humidity generator. This "in the system, as a system" technique helps eliminate systemic errors that might be introduced 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:

$$Y = LIN \times X^2 + SPAN \times X + ZERO \quad (1)$$

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

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, all you need to do is 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 both displayed and stored in the system's NVRAM until the next calibration.

You should run intercomparison validations on a regular basis, tests that compare your equipment with a chilled-mirror hygrometer, psychrometer, or other known consistent humidity-measuring device. Use a variety of humidity values and temperatures, and keep current control charts on all the results to make sure they are within the estimated uncertainty. These charts should include normal trends and abnormalities because this is the record you will use to discover if temperature probes or pressure transducers begin 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 established a basic tracking schedule. Doing this should be part of your overall preventive statistical process control to catch abnormalities before they can cause problems.

Field Trials and Test Data

An RH uncertainty analysis was conducted on the Model 2500 humidity generator, following NIST Guideline 1297. The RH in a two-pressure humidity generator of this type is determined from temperature and pressure measurements using the following formula:

$$RH = P_C / P_S \times E_S / E_C \times F_S / F_C \times 100 \quad (2)$$

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 focused on analyzing the above ratios, both separately and combined, in four specific categories of uncertainty: contribution from the pressure ratio term P_C/P_S, from the vapor pressure ratio term E_S/E_C, from the enhancement factor ratio F_S/F_C, and from saturator efficiency. It was conducted to validate performance accuracy using temperature and pressure uncertainty calculations. Those of the Model 2500 two-pressure humidity generator established 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 at www.thunderscientific.com.

Application Examples

Portable two-pressure humidity generation calibration equipment is widely used in the pharmaceutical, aerospace, and semiconductor industries. It's also the first choice of sensor manufacturers. Let's look at a few specific examples.

Chart Recorders. 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. On-site calibration eliminates unnecessary handling and exposure of the recorder to temperature and/or humidity extremes.

Chilled Mirror Hygrometers. A humidity computer can be used to determine either the saturation pressure or RH necessary to generate a specific dew or frost point. The generator is first 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, then through the chilled mirror head, and finally 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 be placed in the chamber and exhausted to ambient by the slight positive pressure. A flowmeter should be used downstream and adjusted either with a valve or by partial restriction of normal chamber exhaust.

>> Every humidity-generating process requires precise temperature control (set point) and good temperature stability.

Environmental Testing. 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 electronics, plastics, composites, film, and optical equipment, as well as for blood gas analysis and soil hydrology. Depending on the temperature and humidity being generated, the system may operate continuously from hours to months; the only limiting factor is typically the one-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.

Summary

Increasingly stringent testing and calibration will be needed to meet the demands of new technologies being developed for a wide range of instrumentation and devices. Two-pressure humidity generation is proven and traceable to NIST standards, and the portable equipment integrating the technology meets 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 your toughest specification, just as it did when it was first delivered to your lab. ■

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