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A Simple Humidity Generator for Relative Humidity Calibrations

H. G. Liedberg · M. R. Mnguni · D. Jonker

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Abstract At present, the South African national humidity measurement standards, maintained by NMISA, consist of two chilled-mirror hygrometers operating from (−75 to 20) °Cdp and unsaturated salt solutions covering the range (5 to 95) %rh. To reduce measurement uncertainties and to obtain traceability to local measurement standards for temperature and pressure, it is desired to replace the salt solutions by a dew-point generator for relative humidity calibrations from (5 to 95) %rh (5 to 60) °C, equivalent to a dew-point range of (−30 to 59) °Cdp. Required uncertainties in relative humidity (at a coverage factor of $k = 2$) are (0.1 to 1.2) %rh. This article describes the design and evaluation of a two-pressure humidity generator intended to satisfy this requirement. The saturator consists of a coil of stainless steel tubing immersed in a 70 l stirred water bath. Pressure reduction is accomplished using a throttling valve adjusted by a stepper motor, and hygrometers being calibrated are sealed into a small test chamber contained in a larger temperature-controlled chamber. The results of the following performance tests are presented:

(i) comparison of the output when the air stream was oversaturated before entry to the saturator to that when it was dry,
(ii) comparison with a chilled-mirror hygrometer from (−25 to 20) °Cdp, and
(iii) comparison with relative humidity hygrometers that had previously been calibrated against salt solutions.

Keywords Calibration · Dew point · Humidity generator · Relative humidity

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H. G. Liedberg (✉) · M. R. Mnguni · D. Jonker
National Metrology Institute of SA, Private Bag X34,
0040 Lynnwood Ridge, Pretoria, South Africa
e-mail: hliedber@nmisa.org

1 Introduction

The NMISA Humidity Laboratory currently uses unsaturated salt solutions (certified by an accredited European calibration laboratory to uncertainties of (0.1 to 1.2) %rh) as the starting point for traceability in relative humidity, as this allows smaller uncertainties to be achieved in the calibration of relative humidity hygrometers ($U(k = 2)$= (0.4 to 1.4) %rh) than are attainable using the chilled-mirror hygrometer available to the laboratory (part of a temperature- and humidity-variable chamber) as the reference standard [1]. There are several reasons to develop a humidity generator as a reference standard to supplement (and, eventually, replace) the salt solutions:

(i) With gradual improvements in generator design, it may be possible to reduce the uncertainty of this reference standard below that of the salt solutions, allowing more accurate evaluation of hygrometer characteristics, such as nonlinearity, and more accurate calibration of secondary salt solution standards.
(ii) Although temperature coefficient data for the unsaturated salt solutions are available from (0 to 100) °C, relative humidity calibrations are presently limited to the temperature range from (10 to 30) °C, owing to the unknown uncertainty in the salts' temperature coefficients. A reference standard allowing relative humidity hygrometers (RH meters) to be calibrated from (5 to 60) °C would permit more comprehensive characterization of these instruments.
(iii) Relative humidity calibrations will be traceable to local temperature and pressure measurement standards, removing the need to import traceability from another NMI.

Two humidity generators have previously been built at NMISA. The first [2] used a nickel-plated aluminum saturator block that corroded rapidly in water. The second [3] (not intended to be a reference standard) used flow mixing to extend its range to lower dew points, with humid air provided by the same saturator block as the first (for high dew points) and by a permeation tube (for low dew points): although stabilities of (±0.05 to ±0.01) °Cdp were achieved at some dew points in the range from (−60 to 15) °Cdp, the generator exhibited instability of the order of ±0.2 °Cdp when there was a low flow rate through the saturator or when both saturator and permeation tube were used.

The generator described in the present article uses a saturator coil made of 25 mm diameter stainless steel tubing, reducing the possibility of corrosion that affected the first saturator block. It makes use of pressure reduction rather than flow-mixing to generate low dew points, thereby avoiding instability caused by inadequate control of flow rates.

2 Description of Generator

2.1 Design

A schematic diagram of the generator is shown in Fig. 1. Clean, dry air regulated at approximately 700 kPa (absolute) is supplied to the mass flow controller (MFC), which has a flow range of 0–2 standard liters of nitrogen per minute. During normal

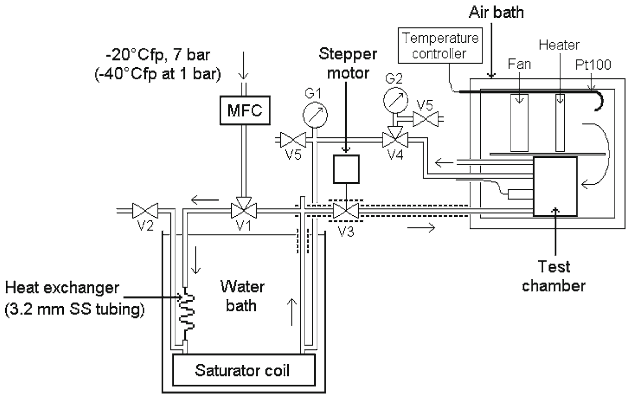

Fig. 1 Schematic of two-pressure humidity generator. Tubing is 6.35 mm diameter stainless steel or perfluoroalkoxy (PFA), except for the heat exchanger (3.2 mm diameter stainless steel) and the saturator coil (25.4 mm diameter stainless steel). Dashed lines indicate heaters wrapped around the tubing

operation, the 3-way ball valve V1 directs the air through a heat-exchanging coil and then through the saturator coil (following the arrows in Fig. 1); however, valve V1 allows the dry air to be flushed backwards through the saturator and vented through needle valve V2, to dry the output line of the saturator (otherwise, water may condense in the output line while the bath is cooling down, later to add excess moisture to the output air when the saturator is operating below ambient temperature) [4,5].

The heat exchanger is a coil of approximately 1.6 m of 3.2 mm diameter stainless steel tubing (inner diameter ∼1.6 mm). The saturator consists of 0.9 m of 25.4 mm diameter stainless steel tubing (inner diameter ∼22 mm); because of difficulties bending this tubing (wall thickness ∼1.8 mm), straight sections of tubing were joined using 90 ° union elbows to form the coil. Approximately 170 ml of distilled water is required to half-fill the saturator. Four 100 Ω PRTs monitor the bath temperature at (100 and 200) mm above the saturator, which is 60 mm above the bottom of the 600 mm deep bath.

A 0–1 MPa (absolute) pressure transducer G1 measures the gas pressure at the saturator outlet. A small bleed-through metering valve V5 prevents the tube to the pressure gauge becoming a dead space, so avoiding back-diffusion of air at the wrong humidity into the air coming from the saturator outlet [4].

A heater is wound around 90 mm of the output line of the saturator where it exits the bath liquid, to prevent condensation in this region (which would otherwise be between saturator temperature and ambient temperature) [4]. The heater wire is electrically insulated with a high-temperature plastic; in addition, it was covered with a section of silicon rubber tubing that was sealed at the bottom with epoxy adhesive. (This epoxy seal later leaked, but the electrical resistance of the heater did not change, indicating that the wire's own insulation is adequate.) 6 W is applied to this heater, raising its temperature approximately 5 °C above the bath temperature. A second heater, electrically insulated with fiberglass, is wound around the section of the output line outside the bath. This heater is controlled at approximately 15 °C above the saturator temperature.

Valve V3 is used to throttle the gas flow, raising the pressure in the saturator above that in the test chamber (which is at ambient pressure). Due to time constraints, adjustment of the valve has not yet been automated using a stepper motor; it is still adjusted manually. V3 was initially a plug valve (1/4 turn from fully closed to fully open), but finer pressure control was achieved using a needle valve (4 turns to fully open). A metering valve with a more finely tapered stem (8–12 turns to fully open) will be tested in the future.

The 300 ml test chamber (50 mm deep) contains ports for relative humidity sensors of three different sizes, two 100 Ω PRTs, and a chilled-mirror hygrometer. In addition, a T-piece in the output line between valve V3 and the test chamber (not shown in Fig. 1) allows connection of a second chilled-mirror hygrometer to the generator output. The test chamber is placed in an air bath of the parallel-tube design, with the working volume 400 mm long × 120 mm wide × 120 mm deep.

A (0 to 250) kPa (absolute) pressure transducer G2 measures the pressure in the test chamber. A bleed valve V5 serves the same purpose as the valve adjacent to G1. The three-way ball valve V4 allows pressure gauge G2 also to measure the saturator outlet pressure (when saturating below 250 kPa).

2.2 Temperature Conditioning and Saturation Efficiency

A total flow rate of $1\,\mathrm{L \cdot min^{-1}}$ (at ambient pressure) is desired, with $0.5\,\mathrm{L \cdot min^{-1}}$ passing through the second chilled-mirror hygrometer (if connected) and the remainder passing through the test chamber. The flow rate in the saturator is highest ($1\,\mathrm{L \cdot min^{-1}}$) when saturating at ambient pressure. (When saturating at 600 kPa, this flow rate is only $1/6\,\mathrm{L \cdot min^{-1}}$.) At $1\,\mathrm{L \cdot min^{-1}}$, the air reaches the saturator inlet approximately 0.5 s after entering the bath and spends 10 s in the saturator. During this time, the air temperature must be lowered by a maximum of 20 °C or raised by a maximum of 40 °C (the water bath is operated at temperatures in the range from (−2 to 60) °C). To test whether the air reaches the correct temperature before leaving the saturator, the temperature of the input air should be varied (perhaps to 10 °C below and above room temperature) when saturating at (−2 and 60) °C, and the effect on the output dew point measured using a chilled-mirror hygrometer [5]. This test has not yet been performed.

The saturation efficiency was evaluated by inserting a Dreschel bottle at 20 °C before valve V1 when the saturator was at (0 and 68) °C, thereby raising the dew point of the input air from −40 °Cfp (150 $\mathrm{ppm_v}$) to close to 20 °Cdp (27000 $\mathrm{ppm_v}$). This should test saturation efficiency adequately at 0 °Cdp (7000 $\mathrm{ppm_v}$), since the mole fraction of the input air is varied from significantly less to significantly more than that generated by the saturator, but may be less effective at 68 °Cdp (330000 $\mathrm{ppm_v}$). It would be preferable to insert a pre-saturator (in a separate bath with independent temperature control) between V1 and the heat exchanger, with valves to switch it in or out of the gas path, so that the input air can be oversaturated at both temperature limits.

3 Results of Performance Tests

3.1 Varying Humidity of Input Air

At a saturation temperature of 0 °C (equivalent to 44 %rh at a test chamber temperature of 12 °C), insertion of the Dreschel bottle before the saturator caused an increase in the generated dew point measured by a chilled-mirror hygrometer of 0.04 °Cdp (equivalent to an increase of 0.1 %rh). At 68 °Cdp (96 %rh at the prevailing temperature of the test chamber), the readings of a resistive electrolyte and a capacitive polymer RH meter decreased by (0.5 and 1.2) %rh, respectively, when the Dreschel bottle was inserted. However, since the generated humidity was fluctuating by ±0.5 %rh because of fluctuations in the saturator pressure, this result may not be meaningful.

3.2 Comparison with Calibrated Hygrometers

The output of the generator was measured by three calibrated hygrometers; a resistive electrolyte RH meter and a capacitive polymer RH meter (both calibrated against unsaturated salt solutions at ambient temperature), and, for dew points below ambient temperature, a chilled-mirror hygrometer with $U(k = 2) = 0.06$ °Cdp. The results are shown in Figs. 2–5.

As the pressure transducers G1 and G2 could not be calibrated before these measurements, the manufacturer's specifications were used as the uncertainties in the pressure measurements ($U(k = 2) = 0.25\%$ of full range, i.e., 2.5 kPa and 625 Pa for G1 and G2, respectively). The uncertainty in saturator pressure is dominant for all measurements except those at the highest saturation pressure (610 kPa), where the uncertainty in the test chamber pressure is dominant. (G1 was used for all measurements of saturator pressure, although G2 could have been used at pressures below 250 kPa.)

Uncertainties in saturator temperature (bath temperature measured by four 100 Ω PRTs) and test chamber temperature (measured by two 100 Ω PRTs) are both estimated to be 0.015 °C; both contribute negligibly to the uncertainty in the generated dew point or relative humidity.

The following observations are made regarding the above results:

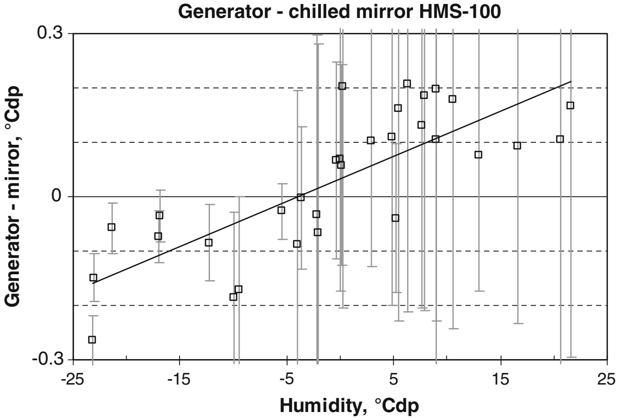

Fig. 2 Measurement of generator output by a chilled-mirror hygrometer. (Uncertainty bars show the uncertainty in the generated dew point, but exclude the chilled mirror's calibration uncertainty of 0.06 °Cdp)

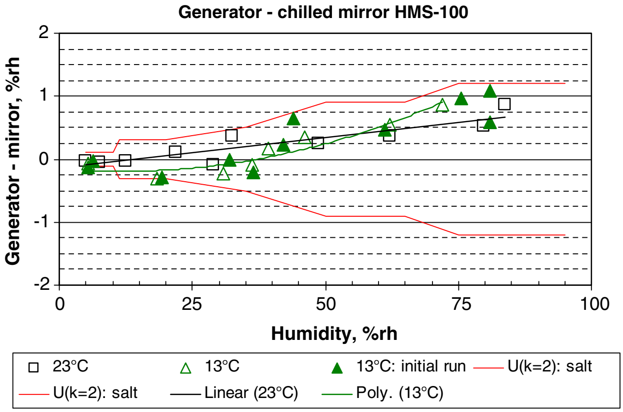

Fig. 3 Measurement of generator output by a chilled-mirror hygrometer, with results converted to relative humidity units (assuming air temperatures of (13 and 23) °C in the test chamber). "$U\,(k = 2)$: salt" indicates the calibration uncertainties of NMISA's unsaturated salt solution reference standards

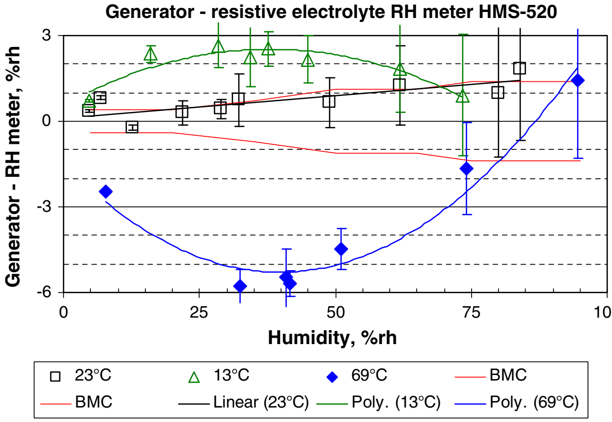

Fig. 4 Measurement of generator output by a resistive electrolyte relative humidity hygrometer. Uncertainty bars show the uncertainty in the generated relative humidity, and "BMC" indicates NMISA's best measurement capabilities for the calibration of RH meters versus unsaturated salt solutions

(i) The disagreement between generator and chilled mirror at high dew points is consistent with incomplete saturation (the hygrometer measures a lower humidity than that predicted from saturator temperature and pressure). The disagreement at low dew points may be caused by the input air being too hot and therefore picking up too much moisture. The temperature conditioning and improved saturation efficiency tests suggested in Sect. 2.2 should be applied. However, the uncertainty in generated dew points is too large to make reliable deductions, because of the large uncertainty in the saturator pressure; the pressure transducers should be calibrated before further testing.

(ii) The RH meter HMS-520 (Fig. 4) has exhibited instability of the order of (2 to 3) %rh during its last two calibrations; the data of HMS-530 are more

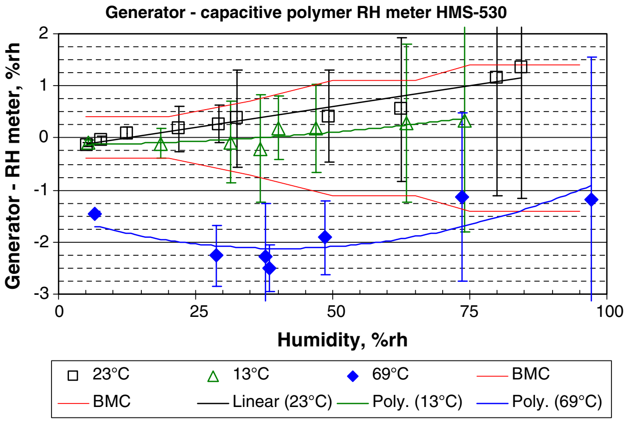

Fig. 5 Measurement of generator output by a capacitive polymer relative humidity hygrometer

meaningful. The results at (13 and 23) °C agree with the hygrometer's last calibration within NMISA's BMCs, but the same trend suggesting incomplete saturation is observed as was seen with the chilled mirror. Again, the uncertainties of the present measurements are too large to draw definite conclusions. As the temperature coefficients of the RH meters have not been measured up to 69 °C, the results at this temperature do not necessarily reflect generator characteristics.

4 Future Development

In addition to the improvements suggested above, the following modifications will also be implemented:

(i) The accurate adjustment of saturator pressure is difficult, and the resultant pressure is not always stable; the use of a fine metering valve to throttle the gas flow will be investigated. Although the minimum achievable saturator pressure may be somewhat higher using such a valve (say, 120 kPa instead of 88 kPa) because the valve offers significant resistance to air flow even at the fully open position, increasing the saturator temperature by a few °C will circumvent this drawback.

(ii) At present, it takes 45 min to raise the temperature of the test chamber by 20 °C and 3 h to lower its temperature by 10 °C; a copper coil circulating chilled water between the air bath's fan and heater, as well as a Peltier element attached to the base of the test chamber, may help to accelerate temperature changes. (The Peltier element should, of course, be switched off before starting measurements.)

(iii) More data comparing the generator output to the readings of calibrated hygrometers are needed; a chilled-mirror hygrometer capable of operating to 60 °Cdp should be installed in the test chamber together with three calibrated relative humidity hygrometers for these tests.

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