

DESIGN, CONSTRUCTION AND EVALUATION OF A HUMIDITY GENERATOR

Amaury Roteger de Oliveira
 Renato Nunes Teixeira

Instituto Nacional de Metrologia, Qualidade e Tecnologia - Inmetro
 Diretoria de Metrologia Científica e Industrial – Dimci - Divisão de Metrologia Térmica – Diter – Prédio 4
 Av. Nossa Senhora das Graças, 50 - Xerém – Duque de Caxias – RJ - CEP 25250-020
 e-mails: aroteger@inmetro.gov.br rnteixeira@inmetro.gov.br

Abstract. Humidity measurement is very important for many technological areas such as pharmaceutical laboratories, food processing, textile and paper industries and for commercialization of grains. The development of a dew-point generator at Inmetro will place Brazil at the same level of a selected group of countries that has this type of technology, enabling the maintenance and dissemination of this quantity at low uncertainties.

The objective of this paper is to present the initial results of the evaluation of a humidity generator developed and constructed at the Hygrometry Laboratory of Inmetro. An air or dry gas flow is purged through the equipment, which humidifies this flow ideally to its saturation point. The equipment is capable to work in the dew point temperature range from $-60\text{ }^{\circ}\text{C}$ to $+15\text{ }^{\circ}\text{C}$, with an estimated uncertainty of $0,10\text{ }^{\circ}\text{C}$ ($k=2$). The main points taken into account during the design and construction phase of this equipment are presented, together with the results of performance tests along the dew point temperature working range, including the uncertainty evaluation. These results are compared to the ones from other Metrology Institutes which developed their own humidity generators. Improvement points for a second version of the equipment are suggested in order to have, in the near future, a primary standard humidity generator, which will make Inmetro independent from external calibration of your standard reference dew-point hygrometers.

Keywords: humidity generator, dew point generator, humidity, saturator

1. INTRODUCTION

The primary reference quantity for the measurement of humidity in air is the mass ratio between water and air, expressed in %. The primary realization of this quantity is performed using a gravimetric system, where the masses from water and air are measured independently. This system of humidity reference is very complex, expensive and takes a long time to perform a measurement. Very few metrology institutes in the world have this kind of facility. Due to these inconveniences, such devices are rarely used in the calibration of hygrometers, being used mostly to evaluate the performance of humidity generators or high quality humidity sensors. Most National Metrology Institutes rely on humidity generators or dewpoint generators as a reference to calibrate humidity sensors (Actiset *al.*, 1998).

Humidity is a physical quantity that is very important for many different areas, such as industry, agriculture and commerce. Also of great importance is the role of humidity in the realization of other physical quantities like length and many optical quantities, which depend on the accurate determination of humidity to correct the refraction index of air, for example. A reliable system of weather forecast is based on precise measurement of humidity and temperature, among other quantities.

On the other hand, in many industrial activities the control of humidity affects directly the quality and cost of a product or service. The measurement and control of humidity are relevant aspects for comfort and safety of the human life. The development of a humidity generator can also benefit scientific research of new humidity sensors, which can use different techniques such as microwaves, infrared radiation or electrical properties of materials.

The construction of this prototype of humidity generator is the first step in towards the independence of Inmetro from other National Metrology Institutes in the calibration of its reference dewpoint hygrometers. This equipment is designed to work in a relatively wide dewpoint temperature range, from $-60\text{ }^{\circ}\text{C}$ to $+15\text{ }^{\circ}\text{C}$. The experience acquired in the development of this device can be used in the future, in the development of improved versions suitable for higher dewpoint temperatures up to $+80\text{ }^{\circ}\text{C}$ or lower than $-60\text{ }^{\circ}\text{C}$ and with lower calibration uncertainties.

This work will detail the construction of the humidity generator, initial results which are compared to those from a calibrated dewpoint hygrometer, an estimate of the calibration uncertainty and also some performance numbers from National Metrology Institutes in Europe that developed their own humidity generators.

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2. SINGLE PRESSURE GENERATOR DESIGN

A humidity generator can be defined simply by an open adiabatic system in which its control volume is maintained at a defined temperature and there is a layer of water in thermal equilibrium. Humid or dry gas flows through this volume, achieving equilibrium by mass and heat transfer, and leaving the equipment at saturation point. Above 0 °C this point is called dew point and below 0 °C generally it is called frost point.

Humidity generators can be divided in two groups: single and two pressure generators. Typically two pressure generators are faster in response, but it is more difficult to control all the influence quantities, resulting in larger uncertainties (Hudoklin, 2008). Single point generators, on the other hand, need larger stabilization time, but can achieve generally lower uncertainties than other type. In this type of generator the dry gas enters the saturator, which is kept at constant temperature T_s and pressure P_s . After leaving the generator, this gas has its dew point temperature T_d measured by a dew point hygrometer. If the pressure at the dewpoint hygrometer is the same as in the saturator ($P_s=P_h$), then the gas temperature in the saturator is the same as in the dewpoint hygrometer. Equation (1) shows how these quantities are related.

$$e_s(T_d) = \frac{e_s(T_s) \cdot f(T_s, P_s)}{f(P_h, T_d)} \cdot \frac{P_h}{P_s} \quad (1)$$

e_s is the saturation vapor pressure and f is the enhancement factor (Anderson, 1996).

The most important part of the equipment is the saturator, which is object of intense studies, leading to different designs, in order to achieve a flow of saturated gas leaving the generator. Other parts of a humidity generation system can be available commercially, such as thermostatic baths, temperature sensors, air pumps and related accessories. Attention should be paid to the selection of such equipment, mainly the thermostatic bath, which should present very good temperature stability and uniformity. The performance of this equipment will impact directly on the performance of the humidity generator. Also of high importance is the temperature measurement system.

2.1 The saturator

Based on the international and national experience of the team of the Hygrometry Laboratory, which are in constant contact with many National Metrology Institutes and literature (de Groot, 1998) it was constructed a first prototype of a humidity generator, so as to evaluate the performance of the equipment and acquire the necessary experience in this field. The saturator design is based on three stages. The first one, called pre-saturator, is formed by copper tubing in the form of a coil and is located inside the thermostatic bath, and it is shown of left side of Fig. 1. The idea is to approach the gas temperature to bath temperature before entering the saturator itself.



Figure 1 – Pre-saturator (L) and saturator (R)

In Fig. 1 on the right, it is possible to see the saturator, which is made of stainless steel, and is composed by the last two stages, where gas stream meets the water surface. The geometry of the equipment is designed to make the gas flow along almost 1 m of wet surface. Inside the saturator, four wire 100 Ω resistance thermometers were installed to measure the water and gas temperatures.



Figure 2 – View of the internal part of the humidity generator

Figure 2 shows the path the gas is forced through the humidity generator. This equipment has two stages, similar to the one shown.

2.2 Experimental setup

In Fig.3 it is shown the complete experimental setup. Dry gas, nitrogen, flows first to the pre-saturator and then to the saturator, where the temperatures of the gas and water are measured using the temperature sensor, which are connected to a digital thermometer. This instrument makes the measurements remotely through a computer. After leaving the saturator, the saturated gas has its dew point temperature measured by a dew point hygrometer and then flows to the ambient. In this initial stage, this hygrometer was used to verify the performance of the generator. Normally, this equipment will be the instrument under calibration.

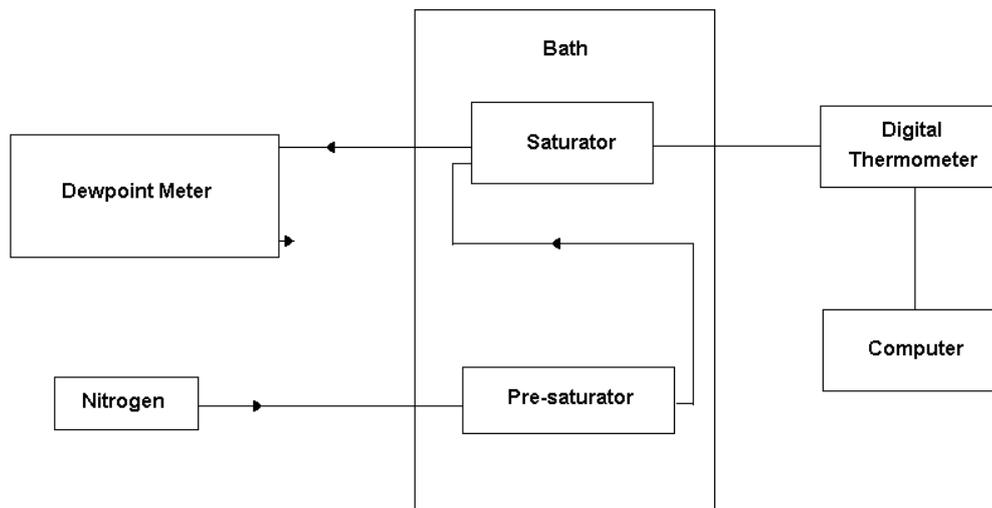


Figure3 – Schematic design of the humidity generation system

The thermostatic bath used in this evaluation was a model 7081 from Hart Scientific. This equipment works in a temperature range from $-80\text{ }^{\circ}\text{C}$ to $+110\text{ }^{\circ}\text{C}$, and had its temperature distribution and stability previously evaluated. The temperatures were measured by $100\ \Omega$ platinum resistance thermometers in four different locations inside the generator, two in the water and two in the gas. These temperature sensors were connected to a digital thermometer from Hart Scientific, model 1560, which was connected to a computer, running dedicated software in charge of the data acquisition, developed at Inmetro. The measurement of the dew point was performed by a dew point hygrometer model S4000 from Michell Instruments, calibrated by the National Physical Laboratory (NPL-UK) in 2011. The nitrogen gas was commercial grade with a purity of 99,999%.

3 RESULTS AND UNCERTAINTY EVALUATION

The first tests performed were to check for gas leakages and also to verify the performance of the humidity generator in various gas flow rates. The leak test revealed that the equipment is gas tight for pressures slightly above normal atmospheric pressure at $15\text{ }^{\circ}\text{C}$, while the results of the flow test can be seen in Tab. 1.

Prior to the beginning of the humidity generation tests, the saturator was cleaned with isopropyl alcohol and distilled and deionized water was used to fill the saturator.

Table 1 – Flowtestresults

Gas Flow	Dewpoint Mean Temperature	Standard Deviation of the Mean	Maximum Temperature Difference
(cm³/min)	(°C)	(°C)	(°C)
200	14,66	0,02	0,08
400	14,67	0,02	0,08
600	14,66	0,01	0,05
800	14,66	0,02	0,08
1000	14,65	0,02	0,07

It can be concluded that for the flow range between 0,2 to 1,0 cm³/min, the performance of the generator was not affected, and we can also conclude that the length of gas in contact with water was long enough to reach the dew point. Typically dew point hygrometers need a gas flow around 0,5 cm³/min, which enables the present humidity generator to calibrate up to two dew-point hygrometers simultaneously, with no penalty in its performance.

The bath temperature was kept at 15 °C and the flow was varied from 200 cm³/min up to 1000 cm³/min, in 200 cm³/min steps. The results shown are a mean value from 30 readings of dewpoint temperature.

Another point evaluated was the temperature difference between the temperatures measured in the air and in the water. Table 2 shows these results, where “dif” is the maximum temperature difference in the 30 readings between water and air temperatures.

Table 2 – Temperature differences between water and air

T/°C	dif/°C
-60	0,017
-50	0,014
-40	0,009
-30	0,003
-25	0,004
-20	0,004
-15	0,005
-10	0,008
-5	0,024
1	0,019
5	0,020
10	0,037
15	0,049

It can be noted in the temperature range between -30 °C to -10 °C, that water and gas temperatures are very close, which was expected. This agreement was not so good at the extremes of the temperature range. This fact is still being investigated to be corrected in a future new version of the humidity generator.

The humidity generator had its performance checked by the comparison of its results with those from the reference dewpoint hygrometer Michell S4000. This verification was made between -60 °C to +15 °C of dewpoint temperature. In this check, the flow was adjusted to 500 cm³/min and 30 readings were taken at every temperature. These results are shown in Fig. 4, where the temperatures of all four sensors were considered.

The comparison curve obtained was: $y = 1,0112x + 0,1349$ with $R^2 = 0,9997$.

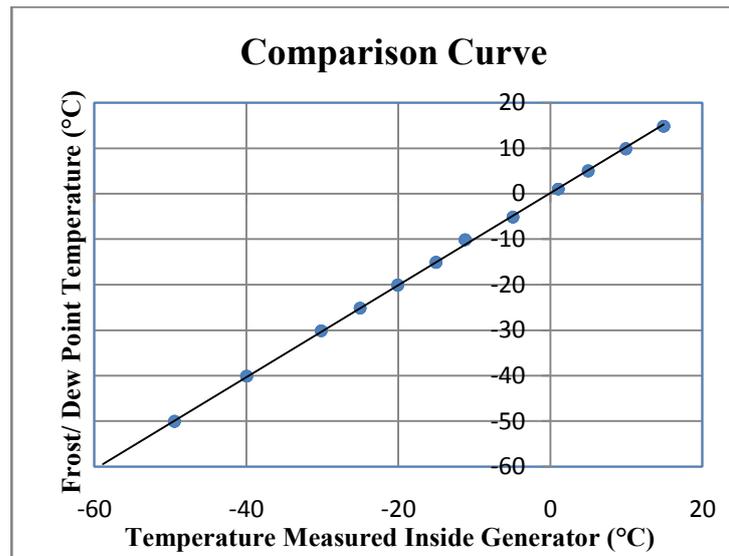


Figure 4 – Comparison check of the humidity generator with the standard dewpoint meter S4000

Differences between the temperatures measured in the humidity generator and in the dewpoint hygrometer were calculated. It can be noted that between -40 °C and $+15\text{ °C}$ these differences were of the order of $0,1\text{ °C}$, which is close to the uncertainty of the dewpoint temperatures of the humidity generator. Below -40 °C these differences reach values close to $+1,1\text{ °C}$ at -60 °C , which is an indication of a limitation of the constructed humidity generator. It is also possible that the plastic tubing used between the outlet of the generator and the dewpoint hygrometer may have absorbed or adsorbed moisture.

In the uncertainty evaluation the following components were considered: uncertainty of the calibration of the temperature sensors (CT), standard deviation of the readings of dewpoint temperatures (SD), resolution of the digital thermometer used in the temperature measurements (RT), temperature gradients (BG) and temperature stability (BS) of the thermostatic bath, maximum temperature difference between water and gas sensors (MD) inside the humidity generator. Table 3 shows an estimate of the uncertainty evaluation for -60 °C of dewpoint temperature. The same model applies for other values of temperature

TABLE3–Uncertainty budget for -60 °C

Source	Uncertainty	Quantity	Distribution	Divisor	Contribution
CT	0,001	°C	normal	2	0,0005
SD	1,00E-03	°C	normal	1	0,001
RT	0,0001	°C	retangular	3,4641	2,88675E-05
BG	0,005	°C	retangular	1,7321	0,002886751
BS	0,0054	°C	retangular	1,7321	0,003117691
MD	0,049	°C	retangular	1	0,049
				u	0,049
				U (k=2)	0,098

The results shown on table 3 are a first estimate, because influence quantities are still be evaluated and were not considered in this approach. These influence quantities are the pressure drop along the gas flow, pressure stability in time, adsorption and desorption of humidity in the tubing and time response of the measuring instruments.

According to Nielsen& Groot (2004), the typical uncertainty of a primary standard humidity generator is around $\pm 0,04\text{ °C}$ ($k=2$). The target of this project was to reach in this stage, uncertainties better than $\pm 0,10\text{ °C}$ ($k=2$) in the measurement of dewpoint temperatures in the range from -75 °C to $+15\text{ °C}$. In Table 4 (Heinonem, 2002), it is shown a summary of the frost/dew point temperature in a comparison of humidity standards between seven European National Metrology Laboratories in 2001. It is possible to see that the target uncertainty and dewpoint range are in line with some laboratories, but there is still space for future improvements.

TABLE 4 – Dewpoint range and uncertainty of seven European National Laboratories

Country	Range (°C)	Standard Uncertainty (k=2) / (°C)
Denmark	-60 to +20	0,13
Poland	-60 to +75	0,033 to 0,064
Switzerland	-60 to +60	0,016 to 0,042
Finland	-40 to +75	0,025 to 0,040
United Kingdom	-60 to +15	0,015 to 0,028
Turkey	-30 to +60	0,16 to 0,25
Russian Federation	-60 to +20	0,050 to 0,054

3 CONCLUSIONS

A humidity generator was designed, constructed and evaluated at Inmetro. The first results indicate that the performance of the equipment is good for the dew-point range temperature from -60 °C to +15 °C and for the flow range from 0.2 l/min to 1.0/min. The performance of the equipment at lower dew-point temperatures (<-60 °C) was not as good as at other dew-temperatures.

Improvements over this first prototype will be implemented in a second generation humiditygenerator, which will include also a computer simulation of the saturation process, in order to achieve the best efficiency of the equipment.

It is also planned to make an interlaboratory comparison with other National Metrology Institute to check the real performance of the equipment, which can help Inmetro to include this service in the calibration and measurement capability (CMCs) database of the Bureau International des Poids et Mesures (BIPM).

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