NIS PRIMARY DEW/FROST-POINT GENERATOR OPERATING FROM -50 °C TO 0 °C IN DEW POINT TEMPERATURE

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Abstract: This paper describes construction and characterization of a dew/frost-point generator developed at national institute for standards. It is intended to operate in the range from -50 °C to 0 °C. The air flows through a saturator controlled by a regulated flow meter. The reference dew/frost-point temperature was measured by Standard Platinum Resistance Thermometer (SPRT) connected to a resistance bridge. A comparative study between the saturator temperature of the new generator measured by SPRT and the dew/frost-point temperature of a calibrated chilled-mirror hygrometer was conducted. It helped to determine the uncertainty of saturator; this uncertainty was found to be between ± 0.06 °C to ± 0.15 °C at confidence limit of 95%. Several experiments were carried out in the mentioned range. Obtained results gave the confidence that NIS generator could be used as a primary humid air generator.

Keywords: characterization, standard, dew/frost point generator, measurement uncertainty.

1. INTRODUCTION

The measurement of humidity plays an important role in industry, including many sectors as diverse as aerospace, electrical, pharmaceutical, textile and food sectors. The dew-point is the unique parameter describing the saturation of gas stream over water and frost point is that for the saturation over ice [2]. There are several techniques available to generate humidity references by using saturated stream gas with very well known water vapor content to be used in calibration of chilled mirror hygrometers [1]. At National Institute for Standards (NIS)-Egypt, a two-pressure humidity generator model 2500 manufactured by "thunder scientific company" was imported. The generator is mainly intended for calibration of dew-point meters in the range from -20 °C up to +50 °C with uncertainty ±0.16 °C and ±0.10 °C respectively. It is used at NIS for research and calibration of many dew-point meters and humidity sensors. The disadvantage of this generator was that, its uncertainty and working range could not be improved.

A Dew/frost point (DP/FP) generator was thus developed at NIS to establish the national standard for humidity with better uncertainty and to respond to an increasing number of requests for accurate calibration of hygrometers at the range from -50 °C to 0 °C. Against this standard are directly calibrated the chilled-mirror hygrometers used as the transfer standard. The new system enables calibration of nearly all types of chilled mirror hygrometers in wide measuring range. The main element of the generator is the saturator which is immersed in a thermostatic calibration bath. Very important is to avoid the un-controlled condensation of the water vapour in the humid gas circuit.

For uncertainty estimation on the DP/FP measurements, a test was performed to examine the behaviour on various DP/FP temperatures. The deviation was between the points measured by a chilled-mirror connected to the saturator air outlet of the generator and that measured by an SPRT immersed in the thermostatic bath in which the saturator is immersed in.

2. THE GENERATOR

Figure (1) shows a schematic diagram of the DP/FP generator which covers the DP/FP temperature range from -50 °C to 0 °C. The main generator parts are the air compressor, the pressure regulator, the dryer, the saturating system, and the transfer-standard chilled mirror.

![Figure (1). Schematic diagram of the DP/FP generator](image-url)
ambient temperature. The air-stream will be cooled inside a saturator, which is designed to be totally immersed in a thermostatic bath. The bath is regulated at certain temperatures. The saturator’s pipe is made of stainless steel with internal diameter of 5 mm and length 1500 mm. A flow controller valve of the chilled-mirror hygrometer is used to set a suitable flow rate in the range from 20 to 60 L/h. The gas saturation-temperature is assumed to be equal to the temperature of liquid bath. An SPRT is placed in the liquid bath to monitor the temperature. The SPRT is connected to an AC resistance bridge, "Tinsley-Senator Type 5840D" that is especially made for SPRTs with a precision of 0.1 mK.

Before starting the experiment the chilled-mirror hygrometer was first switched on and left for 15 minutes to warm up. The chilled-mirror hygrometer was connected to the outlet of the saturator using a Teflon tube.

The measuring system was started and left for one hour for stability. When DP/FP temperature \( t_{d} \) of chilled-mirror hygrometer, and the temperature monitored by SPRT were showing stability, readings were recorded every 5 minutes. The values taken from chilled-mirror hygrometer are compared to those of the SPRT. The following subsections are describing in details the generator components.

### 2.1. PRE-SATURATOR

To minimize any excess evaporative load in the saturator, a pre-saturator was inserted in the dry gas line coming from the dryer. This unit consists of glass cylinder where the dry air is forced to flow over a surface of water at ambient temperature. It is, then, reasonable to assume that in a few passages all the gas in the generator is brought in contact with the water surface.

### 2.2. SATURATOR

The saturator is the main element of the generator. The saturator pipe is helical-shaped made of stainless-steel with inner diameter of 5.4 mm & thickness 1 mm and a length of 1500 mm as shown in figure (2). This saturator is designed to be totally immersed in the thermostatic liquid bath. The bath used is a "FLUKE Model 7381", it has a temperature stability of 0.006 °C.

The temperature is monitored by means of a calibrated "FLUKE" standard platinum resistance thermometer (SPRT) connected to a "TINSLEY SENATOR, automatic thermometer bridge type 5840". This temperature represents the DP/FP temperature.

### 2.3. CHILLED MIRROR HYGROMETER

![Figure (3). Schematic diagram of chilled mirror](image)

The chilled-mirror hygrometer "model DP30", figure (3), manufactured by "MBW" is for laboratory use, for continuous or spot measurement of the DP/FP temperature of air, gas and gas mixture. Its measurement range is from -60 °C FP to 60 °C DP. The resolution of the DP temperature reading is 0.01 °C. The instrument is equipped with internal flow meter that was used to adjust the sampling flow rate.

The chilled-mirror hygrometer was first calibrated by the two pressure generator maintained at NIS [5].

### 3. RESULTS AND DISCUSSION

To demonstrate the saturating capability of the generator and to assess its overall metrological performance, several experiments were carried out in the DP/FP temperatures -50, -40, -30, -20, -10, 0 °C. At equilibrium, the air can be assumed to be fully saturated and the mean of the temperatures measured in the saturator can be taken as its DP/FP. The measurement of the air temperature in the saturator has been considered to be the reference DP/FP temperature. To enable this generator to be used as a reference in the hygrometry field, with the measurement of only the saturator temperature, an evaluation has been conducted by comparing the air temperature in the saturator with the chilled-mirror transfer standard between -50 °C and 0 °C of DP/FP temperature [3].

At the same time with the temperature reading from SPRT the reading from the transfer standard is recorded. The sampling flow rate through the chilled mirror sensor is set to about 30 L/h. The results of the comparison are shown in figure (4), where six runs have been performed showing the differences between the generator temperature measured by the SPRT and the transfer standard DP/FP temperature, expressed in degree Celsius.
Temperature stability of saturator measured by the SPRT during a test carried out at approximately -20 °C is shown in figure (5).

Figure (5). Temperature stability at -20 °C at one run

The value of the fluctuation shows that the generator is stable for accurate calibration of chilled-mirror hygrometers. The stability time is about 4 hours, and this time is enough to calibrate these types of meters even with those having long response time.

Figure (6) and table (1) show the results obtained at all set points. The results are the averages of six runs for each set point. The error bars represent the expanded uncertainty of the measurements. The measurement uncertainty includes combined standard uncertainty of NIS DP/FP generator [4]. The repeatability of DP/FP generator is 0.004 °C.

Table (1) Results of DP/FP generator

<table>
<thead>
<tr>
<th>Bath Set-point °C</th>
<th>DP/FP temperature measured by SPRT t_{dh} °C</th>
<th>DP/FP temperature measured by chilled-mirror hygrometer t_{dm} °C</th>
<th>Difference t_{dh} - t_{dm} °C</th>
<th>Uncertainty °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>-50.00</td>
<td>-49.9123</td>
<td>-49.83</td>
<td>-0.0823</td>
<td>0.08</td>
</tr>
<tr>
<td>-40.00</td>
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<td>0.0144</td>
<td>0.06</td>
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</tr>
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<td>0.0608</td>
<td>0.09</td>
</tr>
<tr>
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<td>0.15</td>
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<tr>
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<td>0.0587</td>
<td>0.20</td>
<td>-0.1413</td>
<td>0.14</td>
</tr>
</tbody>
</table>

The overall performance of the generator was thus tested by comparison against the chilled-mirror hygrometer which was calibrated by the two-pressure generator maintained at NIS.

The uncertainty shown in the last column in table (1) is the expanded uncertainties at confidence limit of 95% that comprise uncertainties estimated due to statistical and non-statistical methods [6].

The DP/FP uncertainties depend on the following elements:
1- Temperature measurement within the saturator (SPRT, smoothing offset, drift between two calibrations, self-heating at probe level, influence of ambient temperature and repeatability).
2- Humid air generation (circuit sealing, pressure measurement, influence of air flow within the saturator and stability of the thermostatic bath).
3- Measurement of flow.

The total uncertainty is thus a composite value integrating the uncertainties of each element in the standard humidity generator (the expanded uncertainties are equal to twice the standard uncertainties).
4-CONCLUSION

NIS facilities for humidity generation were to cover the range of DP temperature from -20 °C to +50 °C using the two-pressure generator with uncertainty of 0.16 °C to 0.10 °C. To extend the capability for lower range with better uncertainties, the new standard DP/FP generator has been built and characterized. It is capable of measuring the reference DP/FP temperature with the saturator’s SPRT. It covers the range between -50 °C and 0 °C with a measurement uncertainty between 0.02 °C and 0.15 °C. The new generator is now ready to be used, mainly in the calibration of chilled-mirror hygrometers. Its operation is fully automated, which means that chilled-mirror calibrations can be made more efficiently. The results give the confidence that NIS DP/FP generator could be used in an intercomparison between NIS and other National Metrology Institutes (NMIs).

5. REFERENCES