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PRIMARY DEW-POINT GENERATION BETWEEN 1°C AND 60°C AT LPM

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Abstract: EUROMET collaborative project has been set up between Finish Centre for Metrology and Accreditation (MIKES) and Croatian Laboratory for process measurements (HMI/FSB-LPM) with the objective to provide HMI/FSB-LPM with two new primary dew-point generators. Both generators operate on single pressure – single pass principle and together cover the dew-point temperature range from -70°C to 60°C, with gas flow rates up to 2.6L/min.

This article presents the design of the high range generator (HRS), which operates in a dew-point range between 1°C and 60°C. To investigate how close the generator is to the ideal saturation, MIKES conducted several efficiency tests, which will also be described. Results of the tests show that HRS generator is sufficiently efficient for a primary realization of the dew-point temperature scale in the specified ranges. The estimated standard uncertainties due to the non-ideal saturation efficiency are found to be between 0.02 °C and 0.05 °C.

Keywords: dew-point generator; humidity; saturator.

1. INTRODUCTION

Until now, several hygrometer calibration facilities have been developed at the Laboratory for Process Measurement (LPM), which is designated laboratory of the Croatian National Metrology Institute (HMI) and maintains national standards for pressure, temperature and humidity. The first humidity facility was completed in 1997 [1], and it was based on two temperatures re-circulating humidity generator with dew-point temperature range between 5°C and 60°C. Second humidity generator was built in 1999 [2, 3] and had a wider dew-point range (-15°C to 60°C), wider test chamber operating range (-15 to 90°C), better stability, quicker response to stepwise temperature change and lower overall uncertainty.

In year 2006. EUROMET Project no.912. was set up between MIKES and LPM with the aim to provide LPM with new primary dew-point generators extending this way dew-point range of existing facility and improving its uncertainties. The low-range generator was designed for primary realization of the dew-point temperature scale in the range between -70°C and 10°C [4] and the high-range generator in the range between 1 and 60°C. MIKES designed and constructed both generators and conducted initial efficiency tests and LPM took care of purchasing and adapting liquid baths, of implementing the temperature and pressure measurement equipment appropriate for use in the system, of the inlet gas preparation and flow control system as well as of the computer-based for automated data acquisition. Some methods for investigation of dew-point generators are given in [5].

This article presents the design of the high range generator (HRS) as well as methods and results of testing its efficiency.

2. OPERATION PRINCIPLE AND DESIGN OF THE GENERATOR

Generators with different basic operating principles have been constructed and described by several laboratories [6–14]. Following the recent trends in dew-point generator design; LPM adopted the single pressure – single pass dew point generator, i.e. sample gas passes through a saturator only once and its dew-point temperature is controlled only by controlling the saturator temperature.

Generator consists of following main parts:

- heated enclosure,
- initial humidifier,
- presaturator,
- coiled-tube heat exchanger and
- saturator chamber.

Before entering the generator, air is first dried and filtered, to avoid generator contamination, and then led into the initial humidifier, where it is humidified to a dew-point temperature well above the desired saturation temperature. Initial humidifier comprises a partly filled water container placed inside the heated enclosure. The enclosure temperature is controlled with a heating system consisting of fan, heater, thermometer and controller.

After initial humidification, air enters the presaturator, where it is dried to the dew-point temperature slightly above the saturator temperature. The pre-saturator is a simple cylindrical vessel completely immersed into the bath liquid in vertical position, partially filled with water. Low end of the drain tube, positioned in the centre of the presaturator, fixes the water at a desired level. The generator is prepared for use by introducing purified and de-ionized water into the presaturator and saturator.

In the coiled-tube heat exchanger, the air is forced to thermal equilibrium with the saturator bath. This causes a small amount of water vapour condensation in the coil.

After passing the coil, the dew-point temperature of air is about the same as the saturator temperature. Finally, complete saturation is ensured by forcing the air in direct contact with water surface, in the saturator chamber. The saturation chamber is a relatively simple, horizontally positioned, box-like vessel, with one baffle ensuring longer pathway of flowing air over the water/ice surface, which covers the bottom. Two additional tubes entering the saturator chamber are used for fixing water level and enabling pressure measurements in the saturator chamber, respectively.

Saturated air is drawn off from the saturator chamber through the straight vertical outlet tube. Outlet tube passes through the heated enclosure, whereby is slightly heated, preventing this way any condensation in the saturated air. For the same reason, the outlet air tube leaving the enclosure must also be heated.

For determination of the saturator temperature, two PRTs are used and their temperature readings are then averaged. Both PRTs are immersed in bath liquid, through the holes in the cover of the heated enclosure and positioned near the saturation chamber.

To minimize tube wall effects, the tubing in which the sample air flow is made of internally polished stainless steel tubes. The only exceptions are the heat exchanger coils in which rougher surface improves the condensation performance.

A schematic diagram of the high-range generator (HRS) is depicted in Figure 1.

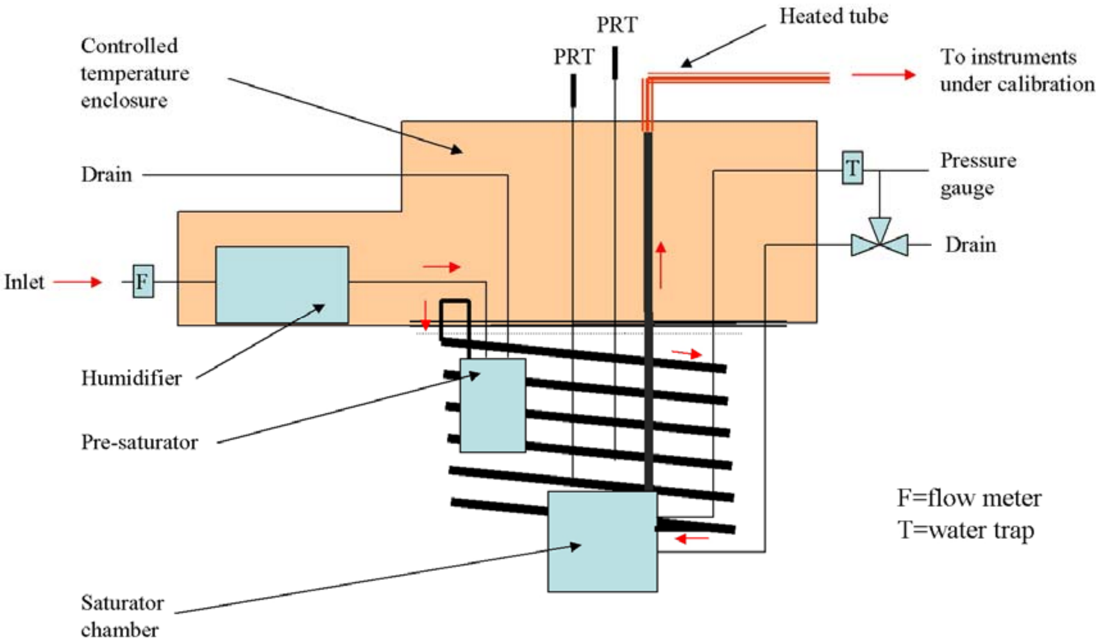

Figure 1. Schematic diagram of the high-range generator

3. TESTING OF THE SATURATOR EFFICIENCY

The saturator must be efficient enough to compensate for the difference in dew points between the inlet and outlet gas under a gas flow that is sufficient to supply at least one hygrometer undergoing calibration. This means that the inlet gas must either take up water (humidify), or excess water must condense in the saturator, in order to achieve an outlet dew-point temperature equal to the saturator temperature.

To investigate the LPM's generator efficiency, tests were performed at MIKES at highest and lowest operating temperatures, i.e. at 1°C and 60°C, respectively. Based on the testing results, uncertainty estimations for saturator efficiency and non-ideality were made.

Efficiency of the saturator was studied by monitoring its output with high quality chilled mirror hygrometer, while varying the inlet air flow rates and dew-point temperatures (the flow rate through the hygrometer was kept constant). Furthermore, the efficiency of the combination of the initial humidifier and pre-saturator was also studied.
The non-ideality tests involved assessing:

- the temperature difference between the saturator bath and the air inside the saturator,
- the pressure drop across the outlet tube,
- the minimum flow rate and
- a comparison with the MIKES dew-point temperature standards through calibrated chilled mirror hygrometer.

The generator was tested in a water bath controlled by a Lauda RC25 thermostat. The tests were carried out at the saturator temperatures of 1°C and 60°C with the stability of the bath within ±0.03 °C. Water bath temperature was measured by two metal sheathed PRTs immersed into the bath liquid, close to the saturation chamber. The thermometers were fitted through the thermometers' holes in the bath and enclosure covers. Resistances were measured using an ASL F700 resistance bridge and 10 channel scanner.

Pressure measurements were performed by using a digital barometer Vaisala PTB220. Calibrated rotameters and capacitive dew-point sensor were used for measuring the flow rate and the dew-point temperature of supplied air. An MBW DP3-D-BCS-III was used for measuring the dew-point temperature of air from the saturator and pre-saturator.

In all the test measurements, the dew-point temperature of the supply air was between -70 °C and -55 °C.

During the tests, the thermally controlled enclosure was kept at least 10°C above the bath temperature. For investigation of the temperature difference between the air flowing through the saturator and water which surrounds it, the additional thermometer was inserted into the saturator pressure measurement tube.

Due to the construction of the test facility, the bath temperature was not very stable, which reduced the accuracy of measurements. In addition, vertical gradients at the high temperature point were larger than expected in the bath used at MIKES.

The efficiency of the combination of the initial humidifier and the pre-saturator was investigated by connecting the hygrometer to the pre-saturator through the pre-saturator drain tube. Measurements were carried out with flow rates between 1 L/min and 3 L/min. The pre-saturation efficiency was found sufficient for the low end of the dew-point temperature range, in the whole flow rate range. However, flow rates less than 1.8 L/min showed insufficient pre-saturation at +60 °C, due to insufficient mixing in the initial humidifier. When operating with flow rates larger than 1.8 L/min, the dew-point temperature of pre-saturated air was at least 6 °C higher than the bath temperature. This prevents a possibility of drying too much in any parts of the flow path, but still keeps moderate water condensation rate inside the main saturator.

The generator efficiency was investigated by connecting the MBW DP3-D-BCS-III hygrometer to its outlet, and supplying air to the inlet. For one part of the test, inlet air was dried using a desiccant dryer while for the other part, air was introduced without drying. Connection between the saturator and the MBW was made using an internally polished stainless steel tube, with a heated hose. An additional heated outlet tube was connected in parallel with the MBW to enable varying of air flow through the saturator, and keeping it at the same time constant through the MBW.

Results of the HRS efficiency tests are presented in Figures 2 and 3. The conditions at each numbered period are given in tables 1 and 2. No flow dependence can be identified. It is worth noticing that the bath temperature decreased almost 0.1 °C, during the tests at +60 °C. The disturbance between the areas no 3 and no 4 in Figure 3 was due to a rapid increase in the bath temperature. Furthermore, the deviation in the last part of the +1 °C curve (Fig. 2) was due to a sudden increase in the bath temperature.

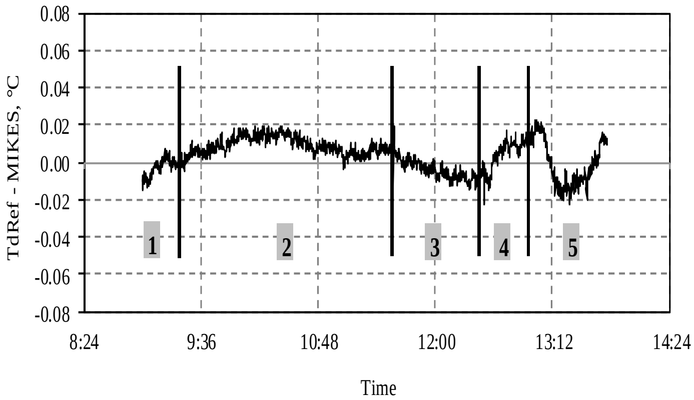

Figure 2. Results of the saturator efficiency tests at 1 °C. The conditions at each numbered period are given in table 1.

Table 1 Conditions of the HRS saturator efficiency tests at 1°C

Test No.	Flow rate through HRS (L/min)	Dew-point temperature of the inlet air (°C)	Temperature of the insulated enclosure (°C)
1	2.3	-60	30
2	2.0	-60	30
3	2.6	-60	30
4	2.6	2	30
5	2.6	1	30

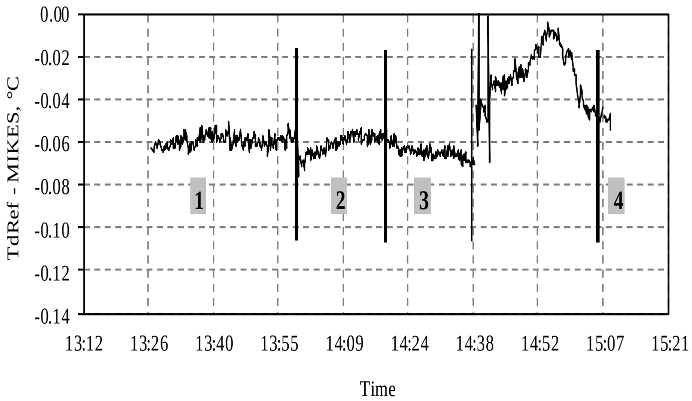

Figure 3. Results of the saturator efficiency tests at 60 °C. The conditions at each numbered period are given in table 2.

Table 2 Conditions of the HRS saturator efficiency tests at 60°C

Test No.	Flow rate through HRS (L/min)	Dew-point temperature of the inlet air (°C)	Temperature of the insulated enclosure (°C)
1	2.3	-60	73
2	2.5	-60	72
3	2.5	3	72
4	2.0	3	71

The results of the tests indicate that the saturator efficiency is sufficient when operating in the saturator temperature range +1 °C to +60 °C, with flow rates (through the saturator) 2 L/min to 2.6 L/min. The standard uncertainty of the saturator efficiency tests was estimated to 0.045 °C.

The temperature difference between the bath water and the air flowing through the saturator was determined by two thin PRTs, one inserted into the saturator pressure measurement tube and other immersed into the bath liquid. However, the heat conduction along the probe in the tube, with zero gas flow, caused an error of about 0.1 °C in the temperature reading, and it was not possible to draw reliable conclusions from the experiment. Error in temperature reading was estimated by comparing measurement results obtained with and without gas flow through the tube, in which the PRT was inserted.

Table 3 summarizes the determined difference between the HRS and the MIKES reference hygrometer.

Table 3 The results of the comparison of the dew-point temperatures determination with the HRS saturator and the MIKES reference hygrometer. (Difference = HRS – MIKES)

Dew-point temperature (°C)	Difference (°C)	Uncertainty (k=2) (°C)
1.40	-0.03	0.13
59.52	-0.04	0.17

4. IMPLEMENTATION OF THE SATURATOR TO THE LPM FACILITY

After initial testing at MIKES, generator was transported to LPM, where it was implemented into humidity facility.

The generator was designed to be used with air as the sample gas, although other gases can also be used. Air is first compressed in an oil-free compressor. In order to decrease pressure variations due to charging and discharging of the proprietary air-tank of the compressor assembly, another much bigger air tank was installed downstream from the compressor. Sample air is then filtered and dried by a twin column pressure swing adsorption dryer and filtered again by additional coalescing filter. This provides particulate-free (down to 10 nm) and dry air at 0.7 MPa gauge pressure and approximately -40 °C frost-point temperature. Air pressure is then gradually reduced to desired level, using three pressure regulators and one small tank. Before entering the generator, air is filtered again, using a 10 nm particulate filter. Air flow rate is monitored by a rotameter. Schematic diagram of the air preparation system is shown in Figure 4.

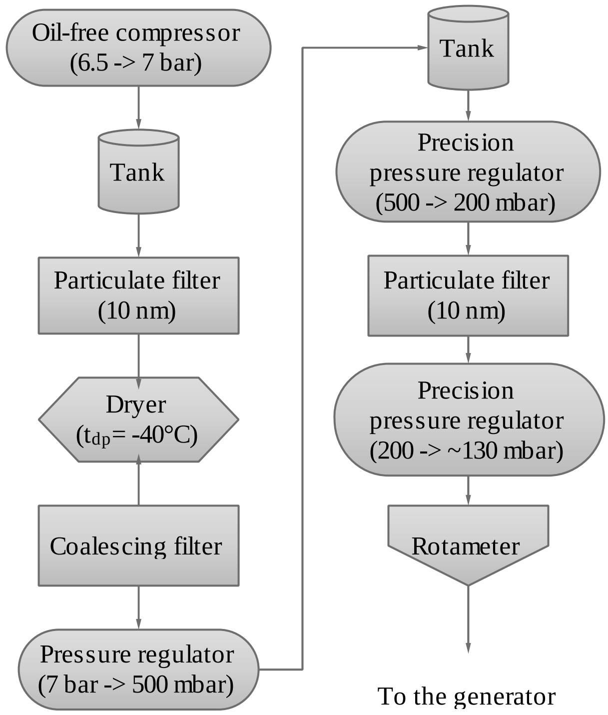

Figure 4. Schematic diagram of the air preparation system

As flowing through the generator, sample air is being saturated to desired dew-point temperature and is then lead to the hygrometer under test. In order to avoid condensation, and thus changing the dew-point temperature of the sample air, all connection tubes between the generator and hygrometer are heated sufficiently above the sample air dew-point temperature. All the connections of the heated tubes to other equipment are also heated. This is accomplished by wrapping insulated heating wire around the connectors and using appropriate temperature controlling equipment. If necessary, an additional heated outlet tube can be connected in parallel with the tested hygrometer. This will enable varying of air flow through the generator, and keeping it at the same time constant through the hygrometer. To avoid condensation inside the float-type flow meters, mounted at the end of the measurement line, water content exceeding saturation conditions at room temperature is removed by a condenser. Digital barometer Vaisala PTB330 is used for continuous monitoring of the pressure drop between saturation chamber and tested hygrometer. All the digital measurement equipment is connected to the PC through RS232 or IEEE 488 interfaces. Custom made LabView software enables controlling the equipment as well as collecting, storage and graphical representation of the measurement data. Schematic diagram of the dew-point measurement line is shown in figure 5.

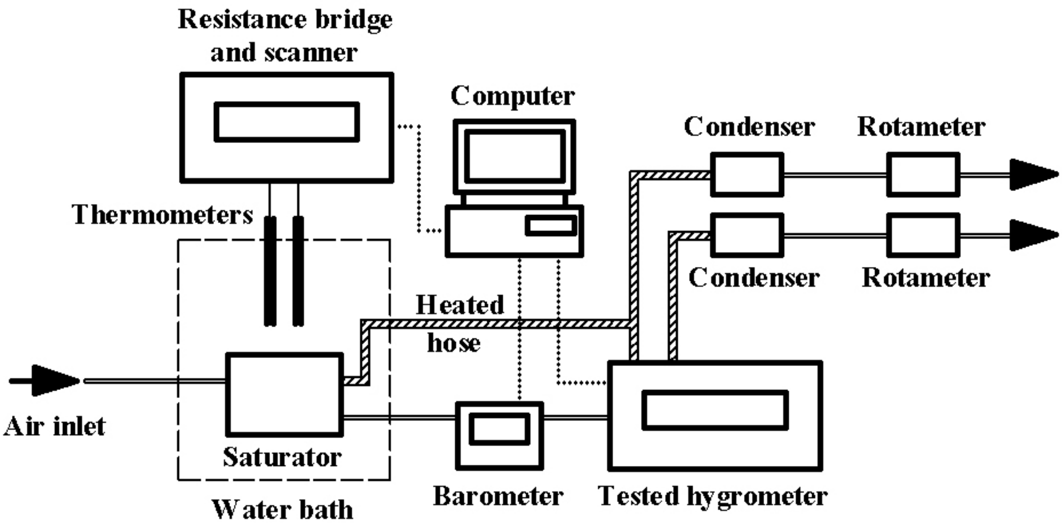

Figure 5. Schematic diagram of the generator implementation at the LPM

5. CONCLUSION

In order to extend the dew-point range and to improve the uncertainties of the humidity scale realization at LPM, the new primary dew-point generator was developed in cooperation with MIKES, starting in year 2006, through EUROMET project no.912. Generator for high dew-point temperature was successfully constructed, tested and applied as the primary humidity standard in LPM.

Results of the efficiency tests show that saturator is efficient enough for a primary realisation of the dew-point temperature scale at gas flow rate which is sufficient for supplying at least one dew-point hygrometer under calibration. The generator covers in the dew-point range from 1 °C to 60 °C under flow rates between 2 L/min and 2.5 L/min. Standard uncertainties due to the non-ideal saturation efficiency are estimated to be between 0.02 °C and 0.05 °C.

Generator is implemented in a dew-point calibration system at LPM, and it is fully operational.

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