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Two-flow system for calibration of hygrometers at room temperature

Master's Thesis

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Table of Contents

List of abbreviations

RH	Relative Humidity
VIM	International Vocabulary of metrology
GUM	Guide to the expression of Uncertainty in Measurement

1. Introduction

Humidity is the presence of water vapor in air. Water vapor is invisible and acts as a gas. The ability of a gas of holding water vapor is strongly dependent on its temperature [1]. As the water vapor is available in our environment, it is in contact with everything we do even without condensing. Vast range of processes are affected by humidity, which includes all kind of manufacturing items that are water sensitive including foods, pharmaceuticals, organic materials [1-5].

As an example in the food industry if the humidity is too high in a food storage the environment is more suitable for accommodating bacteria. And also if the relative humidity is too low, moisture from the food might be extracted causing food quality to deteriorate [1-5].

The trace moisture measuement is critical in the field of semiconductor production because small amount of water vapour can badly affect the performace of semiconductors [5].

It is essential to control the humidity inside buildings such as libraries, museums and hospitals. And also humidity plays a key role in weather and climate change [1, 4-6]. For example, the Fosberg index is based partly on relative humidity and is used to assess the probability of conditions favorable for wildfires. Regarding pollution the size and the scattering coefficient of urban aerosol particles varies directly with the relative humidity [3].

And also humidity is an important parameter for human comfort too. The hot summer days seem to be more hotter due to high humidity. Low humidities can cause human beings to feel dry throat and irritation of eyes. Also the noses of sensitive people may start to bleed due to low levels of humidity [1, 5].

Therefore it is essential to make reliable and accurate measurements in the field of humidity.

Humidity measurements are quite challenging and there are many different measurement techniques [6]. Humidity is expressed using different quantities such as absolute humidity, relative humidity, dew-point temperature, water vapor pressure, mole fraction, etc [1, 5].

Hygrometers are used for measuring humidity. Regular calibration of hygrometers is essential for having accurate and reliable measurement results. Homogeneous and stable humidity environment is critical for the hygrometer calibrations. There are different humidity generating methods such as two-pressure, two-temperature and two-flow methods. These different types of humidity generators are used for calibrating hygrometers. Selection of most suitable humidity generator depends on the measurement range as well as other factors [6, 7].

The aim of this study is to check the suitability of two-flow system for the calibration of hygrometers in the low relative humidity range (2%-30%) at room temperature. The goal includes:

i. Establishment of two-flow system,

ii. Carrying out performance tests in order to assess some important uncertainty components,

iii. Evaluating uncertainty budget for the calibration of reference hygrometers,

iv. Calibrating external hygrometer using established system and its estimated uncertainty.

The existing system for calibration of hygrometers at the University of Tartu does not enable to calibrate hygrometers at very low levels of humidity at room temperature. This study proposes an alternative, reliable and low cost method for calibration of hygrometer in the low humidity range.

2. Literature overview

2.1 Different quantities expressing air humidity

Humidity is the amount of water vapor in air or a gas [1].

There are number of different ways expressing humidity such as absolute humidity, relative humidity, dew-point temperature, water vapor pressure.

Absolute humidity D [g/m^3] is the mass of water vapor in a unit volume of air. In other words this is the mass density of water [1, 8]:

$$D = \frac{m}{V} \qquad (2.1)$$

where m is the mass of water vapor and V is the corresponding volume of the air parcel. The absolute humidity changes with the volume of air parcel that can change with temperature or pressure. Sometimes absolute humidity is called as “water vapor concentration”.

Mixing ratio r_w is the ratio of the mass of water vapor m_w to the mass of air from which the water vapor has been entirely removed m_d in the same air parcel.

$$r_w = \frac{m_w}{m_d} \tag{2.2}$$

Water vapor pressure means partial pressure exerted by the water vapor in a gas [1, 9].

Saturation water vapor pressure is the maximum partial pressure exerted by only water vapor molecules of air that is in equilibrium with plane surface of water or ice at temperature T and pressure P [1, 9].

Enhancement factor f_w is the ratio of saturated water vapor pressure of air E'_w to the saturated vapor pressure of pure water E_w.

$$f_w = \frac{\mathrm{E'_w}}{\mathrm{E_w}} \tag{2.3}$$

Relative humidity RH is defined as the ratio of the actual water vapor pressure e_w to the saturation vapor pressure E_w (with respect to water) at the same temperature and is expressed as a percentage. [1, 8, 9]

$$RH = \frac{e_w}{E'_w} \cdot 100\ \% \tag{2.4}$$

Actual water vapor pressure at air temperature e_w is equal to the saturated water vapor pressure E_w at dew-point temperature or frost-point temperature.

Therefore equation (2.4) can be written as follows,

$$RH = \frac{E_w(T_d)}{E_w(T)} \cdot 100\ \% \tag{2.5}$$

For simplicity enhancement factors are omitted in the above equation.

The dew-point temperature is the temperature at which condensation occurs when a gas is cooled (at constant pressure and composition of air). At dew-point temperature gas becomes saturated with respect to water. It is called the frost-point temperature when gas becomes saturated with respect to ice [1, 8, 9].

2.2 Hierarchy of Humidity measurements

The following figure shows the hierarchy of humidity standards and humidity measuring equipment.

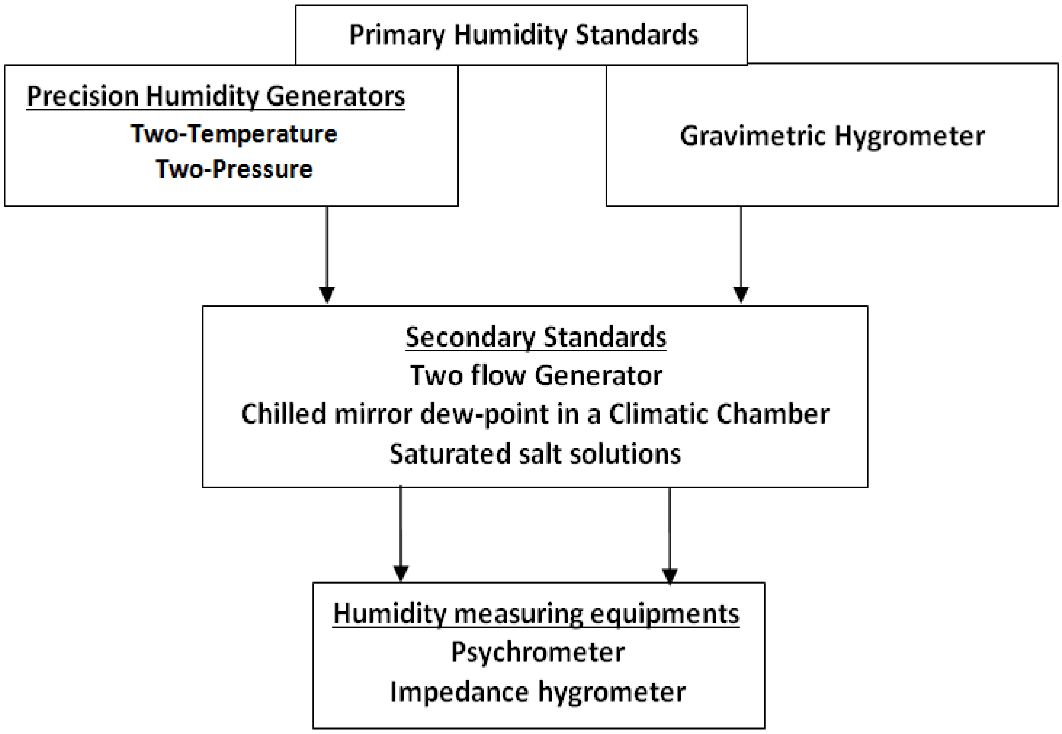

Figure 1. Hierarchy of Humidity Measurements.

2.2.1 Primary Standards

Gravimetric hygrometers measure the mixing ratio of a gas [10]. The mass of the water absorbed by a desiccant is divided by the dry air mass. The dry air mass is calculated by the dry air volume, pressure and temperature.

Mass , temperature, pressure and volume can all be measured with high accuracy. Therefore this system has been considered as the primary standard of humidity measurment [8].

The main drawback of this system is that its operation is time consuming. Therefore it is impractical for calibration service. It is used in a few laboratories to check the performance of primary humidity generators [11,12].

The principal of the two-pressure generator is that air is saturated at a higher pressure and then expanded to a lower pressure at the same temperature. The relative humidity *RH* is calculated using the following formula [7]:

$$RH = \frac{P_c}{P_s} * \frac{F_s(P_s,T_s)}{F_c(P_c,T_c)} \cdot \frac{E_w(T_s)}{E_w(T_c)} \cdot 100\% \qquad (2.6)$$

where P_c is the measurement vessel pressure, P_s is the pressure in the saturator, $E_w(T_c)$ is the saturation vapor pressure of pure water at the vessel temperature, $E_w(T_s)$ is the saturation vapor pressure of pure water at the saturation temperature, F_c is the enhancement factor computed at the vessel pressure and temperature and F_s is the enhancement factor computed at the saturation pressure and temperature [7, 13-15].

In two-temperature generators air is saturated with respect to plane surface of pure water and pumped to the measurement vessel. The temperature of the measurement vessel is maintained higher than the temperature of the saturator. Pressures in the saturator and measurement vessel are maintained almost equal.

The relative humidity in the measurement vessel is calculated in the following way:

$$RH = \frac{F_s(P_s,T_s)}{F_c(P_c,T_c)} \cdot \frac{E_w(T_s)}{E_w(T_c)} \cdot 100\% \qquad (2.7)$$

where $E_w(T_c)$ is the saturation vapor pressure of pure water at the vessel temperature, $E_w(T_s)$ is the saturation vapor pressure of pure water at the saturation temperature, F_c is the enhancement factor computed at the vessel pressure and temperature and F_s is the enhancement factor computed at the saturation pressure and temperature [7,13-15].

2.2.2 Secondary Humidity Generators

The following figure shows the two-flow humidity generator and its main componets.

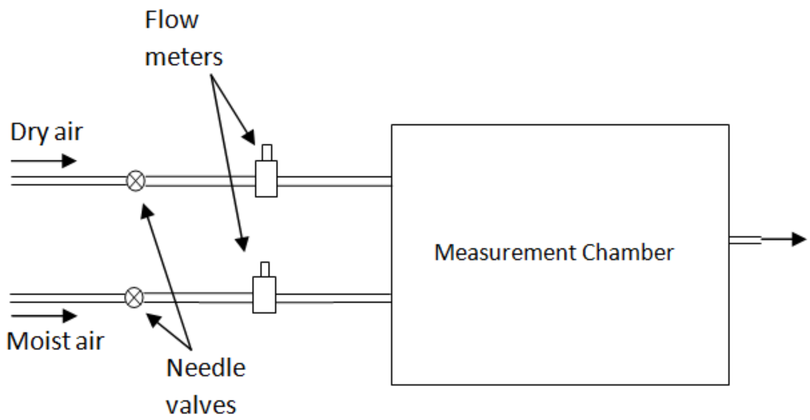

Figure 2. Working principle of a two-flow humidity generator.

In a two-flow humidity generator compressed air is mixed with moist air [4]. It is possible to change the flow rates of compressed and moist air to generate different humidities in the measurement vessel.

The reference value of relative humidity can be calculated by measured flow rates and dew/frost-point temperatues of compressed and saturated air. As an alternative the reference value of humidity (dew/frost-point temperature) can also be measured by an accurate hygrometer (e.g. chilled mirror dew-point hygrometer)[6].

If two-flow system is established with very precise mass flow controllers then it can be considered as a primary humidity standard. In this case main uncertainty sources are due to the generation of saturated air, flow rate measurment uncertainties and air temperature measurement uncertanty in the measurement vessel [6]. Otherwise two-flow system is used as a secondary standard. In this case the main uncertainty sources are due to uncertainty of the reference hygrometer, dew-point temperature stability in the system and temperature measurement uncertainty in the measurement vessel. In this work two flow system is considered as a secondary standard [10,13].

The working principle of chilled mirror dew-point hygrometer lies on cooling a mirror until dew or frost layer forms on it. The temperature of the mirror is measured by an accurate thermometer that is imbedded in the mirror. So the instrument measures either the dew-point temperature or the frost-point temperature of gas. Additionally it is possible to measure air temperature using a separate thermometer. Using these two parameters relative humidity can be calculated. The mirror temperature is controlled automatically and the thickness of the water layer is monitored with a reflected light beam irradiating a light detector [16].

The main advantages of chilled mirror dew-point hygrometers are low uncertainty, high precision, good long term stability and wide measurement range.

Climatic chambers make specified environmental conditions artificially, such as extreme temperatures, temperature variations, relative humidity, and vacuum. In the humidity measurements climatic chambers are used to calibrate hygrometers and they provide stable humidity levels at known temperatures [6].

Very simple and cheap standards that can be used for humidity calibrations are saturated salt solutions. At any temperature the concentration of saturated solution is constant and does not need to be determined. These salt solutions make environments of known relative humidities in small closed volumes [17, 18].

2.2.3 Humidity Measuring Equipment

The working principle of a psychrometer is based on evaporative cooling of wet bulb thermometer with respect to dry bulb thermometer. The rate of evaporative cooling is dependent on relative humidity of air. It is called as wet and dry bulb hygrometer too because it consists of two paired thermometers. One thermometer is sheathed in a wet wick and the other is kept dry. The temperature values indicated by the two thermometers can be used to obtain the relative humidity values using the psychrometer table or corresponding psychrometer formula [8, 12, 16].

This method is simple, cheap, reliable and robust. Psychrometers have good stability and can be used for measurement in wide range of humidity values. Main drawback of this instrument is related to slow response time. Also the contamination of wick around the wet bulb thermometer can cause incorrect results.

Impedance hygrometers are widely used in industry. These can be dividend into capacitive and resistive hygrometers [1, 8, 16]. These hygrometers are constructed from polymer material with a hygroscopic dielectric and are designed to provide an electrical capacity or resistance change corresponding to relative humidity of air. The thin film polymer absorbs water vapor as the relative humidity of the ambient air rises and desorbs water vapor when relative humidity drops.

These instruments are simple and relatively cheap but there can be calibration drifts after experiencing high temperatures or high humidities and these instruments can also be damaged by chemicals.

2.3 Measurement uncertainty

According to the International Vocabulary of Metrology [19] measurement uncertainty is a non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used. In general uncertainty can be introduced as eighther doubt or lack of knowledge about the measurement.

The measurand is the quantity to be measured and the objective of any measurement is to determine the value of the measurand [19]. Measurand can be linked with input quantities using equation and it is called as mathematical model or measurement equation. Measurement uncertainty can be expressed as standard deviation and it is called as standard uncertainty. Combined standard uncertainty is calculated from individual standard uncertainties associated with the input quantities of measurement equation. The product of the combined standard uncertainty and coverage factor is called as expanded uncertainty, which defines an interval around the measured value, where the true value lies with stated probability. The measurement uncertainty itself is the half-width of that interval and is always non-negative. There are two types of uncertainies. Type A uncertainty values are obtained by using statistical methods and type B uncertainty values are obtained by other than statistical methods. Uncertainty is very necessary to check whether the two measurement results agree metrologically to each other. This is called as compatibility of two measurement results [19, 20, 21, 22].

3. Experimental Setup and calibration of hygrometer

3.1 Experimental Setup

A two-flow system for calibration of hygrometers has been set up operating at room temperature.

As shown in figure 3, basically the flows of compressed air and saturated (moist) air are set by the needle valves of the two flow-meters. The mixed air is transferred to the measuring vessel and using three sensors in it temperature is being measured. Relative humidity reference value is calculated from dew/frost-point temperature and air temperature in the measurement vessel. Chilled mirror dew-point hygrometer is used as a reference standard for measuring either the frost-point temperature or the dew-point temperature of the mixed air.

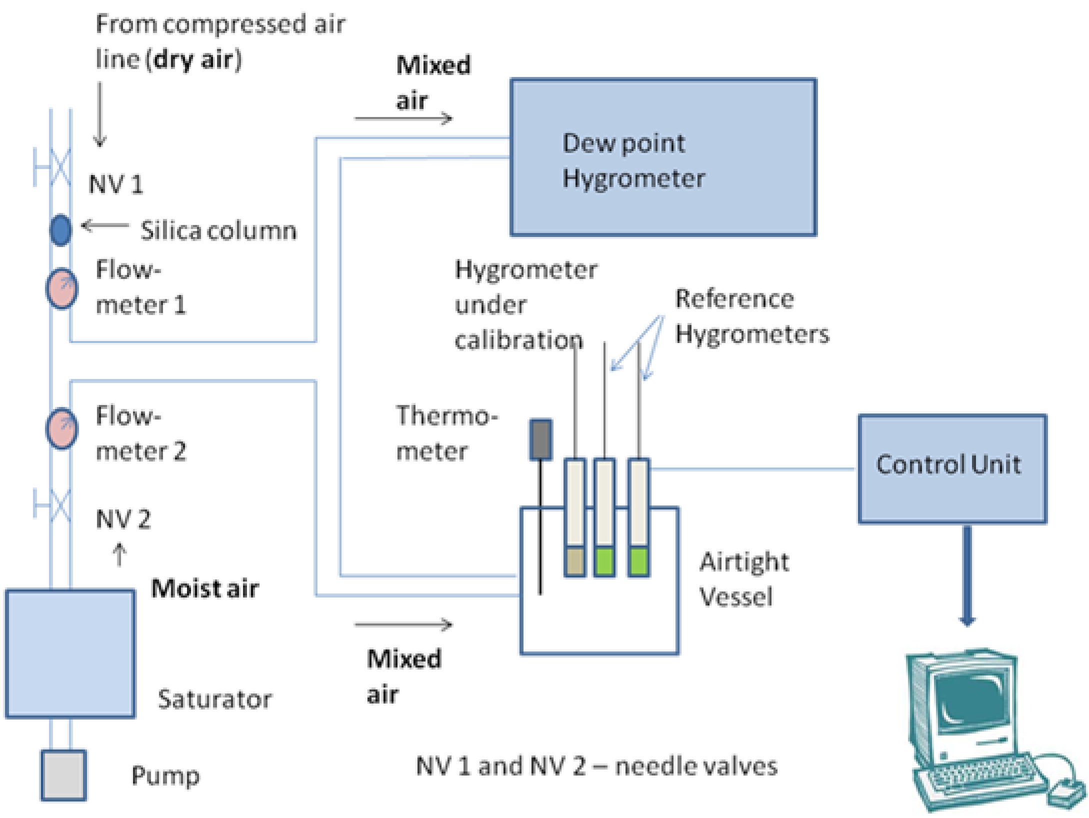

Figure 3. Established two-flow system for calibrating reference capacitive hygrometers.

Compressed air was taken from the compressed air line of Institute of Chemistry and silica gel column was kept in the dry air branch to increase the frost-point temperature stability of dry air. The frost-point temperature of air flowing in the dry air branch was about -40 ºC and for saturated (moist) air the dew-point temperature was set about 17 ºC.

The simplified dew-point generator is used to produce saturated air and its working principle is shown on Figure 4 below.

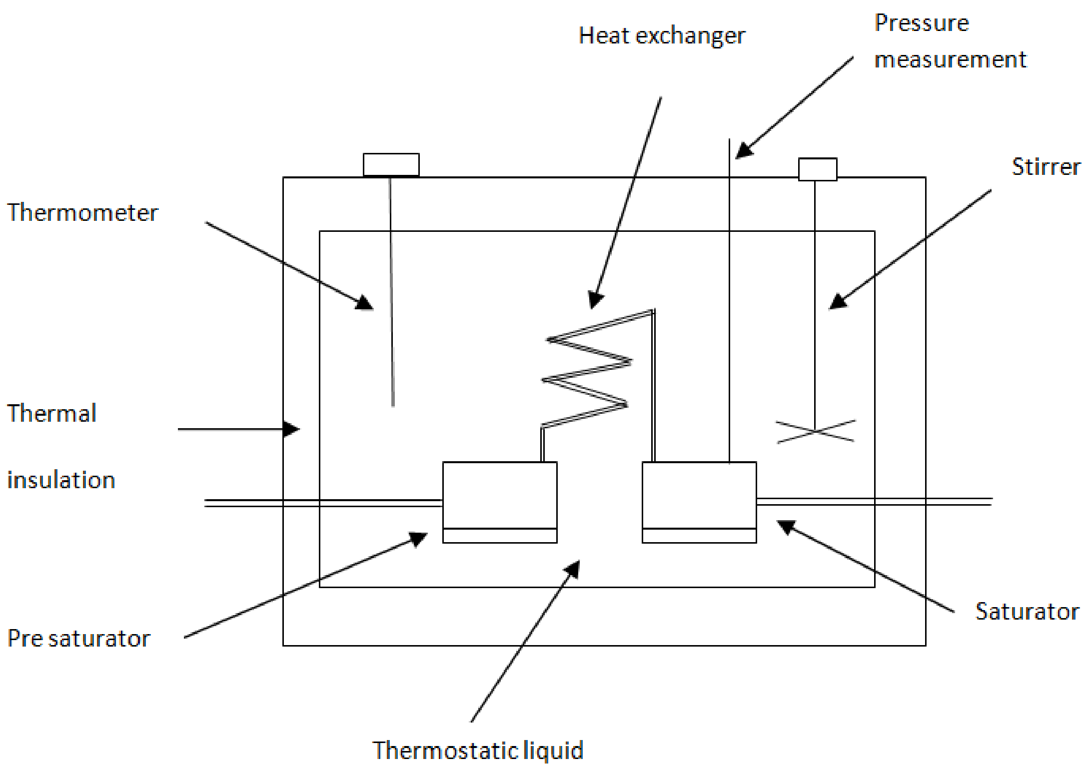

Figure 4. Simplified dew-point generator.

The simplified dew-point generator mainly consists of saturator, heat exchanger and pre-saturator and all of them are immersed in a temperature controlled bath with thermostatic liquid (water) [10]. The temperature of the thermostatic liquid (water) was controlled by thermostat model: U2 and the bath temperature was measured by a calibrated industrial Platinum Resistance Thermometer (IPRT). Water was mixed continuously by using a stirrer. A tube is available in the saturator to measure the pressure in it. This system decreases the dew-point temperature of flowing air from pre-saturator to saturator and makes suitable environment for condensation. Most of the condensation takes place in the pre-saturator and heat exchanger. The final condensation takes place in the saturator. The pre-saturator and saturator vessels have been made of stainless steel mugs.

Needle valves and flow meters were used to set the flow-rates of compressed air and saturated air. Two mechanical flow meters with maximum measuring capability 1.25 L/min and 2.6 L/min were used for adjusting flow rates of compressed air and saturated air in order to obtain different relative humidities in the measurement vessel. These two flow meters were calibrated using gas meter Elster BK-G6T. The system was setup using Teflon tubing.

Mixed air flows through the parallel connection of the chilled mirror dew-point hygrometer and the measurement vessel. Measurement vessel is an oval shape vessel. Its diameter, depth and volume are about 21 cm, 15 cm and 3 L, respectively. It is made of stainless steel. The vessel is closed with an air tight cover with three ports for setting probes in it. It also contains a tube for pressure measurements in the vessel.

Basically, three measuring probes were used for this work. Each probe consists of two sensors. A thermistor was used for air temperature measurement and capacitive sensor was used for relative humidity measurements. Temperature sensors were calibrated in water using climatic chamber and two calibrated Platinum Resistance Thermometers (PRT). During calibration protective caps were used to prevent water from damaging the sensors. The relative humidity sensors were calibrated using the established two flow system. All measuring sensors were connected to multi-channel control unit Almemo 2290-8, which is connected to computer with commercially developed data acquisition and control software called AMR. Temperature and relative humidity values were recorded in every five minutes and they were stored in personal computer.

3.2 Calibration procedure

Two types of calibration works were carried out using this established two-flow system. In the first part the relative humidity sensors of the three reference hygrometer probes were calibrated. In the second part the external hygrometer was calibrated using above calibrated reference hygrometers.

3.2.1 Procedure for calibration of reference hygrometers

The established system was run overnight with compressed air only. Then during the next day series of relative humidity values (2%-30%) were obtained changing the flow rates of saturated and compressed air using the needle valves of the two flow meters. For every relative humidity value the system was run till stable relative humidity was achieved. The relative humidity reading of the reference hygrometer under calibration was directly compared with the corresponding reference value. The reference value of relative humidity was obtained from the dew-point temperature measured by the chilled mirror dew-point hygrometer and air temperature measured by the temperature sensors of the reference probes.

3.2.2 Procedure for calibration of external hygrometers

One of the reference hygrometers was replaced by the external hygrometer (it is also possible to place relative humidity loggers or some types of hygrometers on the bottom of the measurement vessel without removing the third reference sensor) that was needed to be calibrated. The same procedure was followed as for reference hygrometer calibration. The relative humidity reading of the hygrometer under calibration was directly compared with the relative humidity values obtained from the previously calibrated reference hygrometers.

3.3 Performance Tests

In order to carry out hygrometer calibrations, it is very necessary to maintain homogeneous and stable humidified environment in the measurement vessel. The temperature stability of the measurement vessel is also an important parameter in this process. The following tests were carried out in order to be sure that the above requirements are satisfied and to estimate related uncertainty components.

(i)Dew/frost-point temperature stability and homogeneity of

a. compressed air in the measurement vessel
b. saturated air in the measurement vessel
c. mixed air in the measurement vessel

(ii)Temperature stability and homogeneity of the measurement vessel

(iii)Comparison of humidity values between the measurement vessel and the chilled mirror dew-point hygrometer branches

3.3.1 Dew/frost-point temperature stability and homogeneity tests

Dew/frost-point temperature stability was checked for the compressed air, moist air and mixed air separately. During compressed air testing, the system was run overnight only with compressed air supply for removing residual moisture from the inner surface of the tubing and for stabilization. For moist air it was sufficient to run the system for a shorter period. In the case of mixed air the system was run overnight only with compressed air supply. During the next day mixed air was generated controlling the compressed and moist air flows by using two flow meters. Several flow rates of compressed air and moist air were used to achieve the different humidity levels (2%-30%) in the measurement vessel. Each humidity level was maintained with considerable time for stabilization. In the above three cases the chilled mirror dew-point hygrometer was used as the reference and its flow meter reading was maintained 500 ml/min throughout all the experiments by controlling the flow rate of output air stream. The reference probes for measuring relative humidity and air temperature were located triangularly 7 cm apart from each other in the measurement vessel. The data collecting time was set to 5 minutes and all the data were stored in the personal computer. With the help of Sonntag equations the dew/frost-point temperature values were calculated by measured air temperature and relative humidity values. It was possible to estimate the dew-point temperature instability related uncertainties by analyzing the dew-point temperature values obtained by the three probes. Dew/frost-point temperature homogeneity was checked for the mixed air at 2% and 30 % relative humidity levels. In case of 2% relative humidity level the system was run overnight with compressed air. For 30% it was not necessary to do so. After the stabilization, relative humidity and air temperature measurements were recorded for few hours. Then two of the three sensors were interchanged in the measurement vessel. Air temperature and relative humidity readings were recorded as before sensors were interchanged. The swapping of the two sensors helped to cancel out the errors of the two relative humidity sensors to large extent. Calculations were done using Sonntag equations and differences of calculated dew/frost-point temperatures can be used to estimate standard uncertainty of inhomogeneity of mixed air at 2% and 30 % relative humidity levels.

3.3.2 Air temperature stability and homogeneity in the measurement vessel

During the stability and homogeneity tests (3.3.1) and calibration of reference hygrometers (3.2.1) measurement vessel temperature was recorded continuously by using three calibrated temperature sensors of the same reference probes. These recorded results were analyzed and the uncertainties due to temperature instability and inhomogeneity in the measurement vessel were evaluated separately.

3.3.3 Comparison of humidity values between the measurement vessel and the chilled mirror dew-point hygrometer branches

In the calibration of reference hygrometers two parallel branches were used in the system that were connected to the measurement vessel and the chilled mirror dew-point hygrometer respectively as shown in figure 3. During the calibration of reference hygrometers relative humidity values were obtained via chilled mirror dew-point hygrometer readings and measured air temperature values in the vessel. The calculated relative humidity values were directly compared to relative humidity values indicated by the three reference hygrometers in the measurement vessel. In order to make this comparison, it was very necessary to study how equal are the dew/frost-point temperatures in the two branches. For this experiment dew-point temperature was calculated via measured relative humidity and air temperature values by probes located in the measurement vessel. The fourth probe of the same type was inserted in a small stainless steel cylinder in the parallel air flow branch. The volume of the flow path of this small cylinder, similar to the mirror chamber of the chilled mirror dew-point hygrometer, is much smaller than the volume of the measurement vessel. Temperature values as well as relative humidity values were recorded in this cylinder and the corresponding dew-point temperature values were calculated using the Sonntag equations. After few hours of recording the data the measuring probe in the cylinder was interchanged with one of the probes in the measurement vessel and data were collected for few hours again. The swapping of the two sensors helped to cancel out the errors of the two relative humidity sensors to large extent. The dew-point temperature difference between the two branches was calculated by the data of the two measurements (before and after swapping the two probes). The uncertainty component due to the dew-point temperature differences between the two branches was evaluated from the maximum dew-point temperature difference of the two branches.

3.4 Calculations

3.4.1 Mathematical Model

In this study the ultimate goal is to find the relative humidity correction of the hygrometer (reference or external) under calibration. For this purpose following equation can be used,

$$RH_{corr} = RH_{ref} - RH_{mea}, \qquad (3.1)$$

where RH_{corr} is the relative humidity correction of the hygrometer under calibration, RH_{ref} is the reference value of relative humidity and RH_{mea} is the measured relative humidity value of the hygrometer under calibration.

As discussed in the sections 3.2.1 and 3.2.2 two types of calibration works were carried out during this study. When equation (3.1) applies for reference hygrometer calibration, RH_{ref} is the relative humidity value obtained by chilled mirror dew-point hygrometer readings and air temperature readings of the vessel and RH_{mea} is the relative humidity value measured by reference hygrometer (hygrometer under calibration).

For the external hygrometer calibration (section 3.2.2) RH_{ref} is the relative humidity value measured by two or three reference hygrometers which are calibrated in the first part and RH_{mea} is the relative humidity value measured by external hygrometer (hygrometer under calibration).

Relative humidity inside the measurement vessel can be expressed by the following formula:

$$RH = \frac{f(p,T_d)\cdot E_w(T_d)}{f(p,T)\cdot E_w(T)} \cdot 100\ \% \qquad (3.2)$$

where $E_w(T_d)$ is the saturated water vapor pressure at dew-point temperature (T_d), $E_w(T)$ is the saturated water vapor pressure at air temperature (T) in the measurement vessel and $f(p,T_d)$ and $f(p,T)$ are the enhancement factors at dew-point temperature and air temperature, respectively.

As the enhancement factors weakly depend on temperature it is possible to neglect the enhancement factors in equation (3.2) and the approximate formula can be written as follows [23]:

$$RH = \frac{E_w(T_d)}{E_w(T)} \cdot 100\ \% \qquad (3.3)$$

Saturated water vapor pressure is calculated using the Sonntag formula. The generalized Sonntag formula can be written as follows [24].

$$E_w(T) = A_o \cdot (e^{A\,T^{-1} + B + CT + DT^2 + E\,ln\,T}) \qquad (3.4)$$

Where *A, B, C, D* and *E* are the Sonntag coefficients [24] that are shown in Table 1 below.

Saturated water vapor pressure above ice is lower than above super cooled water. Therefore in the Sonntag formula two different set of constants are used for water and ice respectively [24].

Table 1. Numerical values of constants *A, B, C, D* and *E* for water and ice.

Coefficient	Water	Ice	Units
A_0	1	1	hpa
A	-6096.9385	-6024.5282	K
B	16.635794	24.7219	1
C	$-2.711193\cdot10^{-2}$	$1.0613868\cdot10^{-2}$	K^{-1}
D	$1.673952\cdot10^{-5}$	$-1.3198825\cdot10^{-5}$	K^{-2}
E	2.433502	-0.49382577	1

Throughout this study dew/frost-point temperature plays a significant role.

Dew/frost-point temperature can be calculated by the following equation:

$$t_{d/f} = Ay + By^2 + Cy^3 + Dy^4 \tag{3.5}$$

where *A*, *B*, *C* and *D* are coefficients [24] that apply for water, supercooled water and ice. These coefficients are shown in Table 2 below. In equation (3.5) *y* can be calculated by actual water vapor pressure *e* using the following equation:

$$y = ln\left(\frac{e}{a_{d/f}}\right),$$

where, the values of the coefficients $a_{d/f}$ are shown in the last row of table 2 for water, supercooled water and ice.

Table 2. Numerical values of coefficients *A*, *B*, *C* and *D* for water supercooled water and ice.

Coefficient	Dew-point temperature(t_d)		Frost point	Units
	water	supercooled water	ice	
A	13.715	13.7204	12.1197	°C
B	$8.4262 \cdot 10^{-1}$	$7.36631 \cdot 10^{-1}$	$5.25112 \cdot 10^{-1}$	°C
C	$1.9048 \cdot 10^{-2}$	$3.32136 \cdot 10^{-2}$	$1.92206 \cdot 10^{-2}$	°C
D	$7.8158 \cdot 10^{-3}$	$7.78591 \cdot 10^{-4}$	$3.8403 \cdot 10^{-4}$	°C
$a_{d/f}$	6.11213	6.11213	6.11153	hPa

Actual values of air temperature (T) and dew/frost-point temperature ($T_{d/f}$) can be obtained from the following equations:

$$T = T_{mea} + \Delta T_{inhom} + \Delta T_{cal} + \Delta T_{res}, \tag{3.6}$$

where T_{mea} is the average temperature reading indicated by the temperature sensor, ΔT_{inhom} is the correction due to temperature inhomogeneity, ΔT_{cal} is the correction of the temperature sensor due to its calibration and ΔT_{res} is the correction due to the resolution of the temperature sensor. In equation (3.6) the values of ΔT_{inhom} and ΔT_{res} are assumed to be zero.

$$T_{d/f} = T_{d/f\,mea} + \Delta T_{d/f\,inhom} + \Delta T_{d/f\,cal} + \Delta T_{d/f\,res} + \Delta T_{d/f\,dtb}, \tag{3.7}$$

where $T_{d/f\,mea}$ is the average dew/frost-point temperature reading indicated by the chilled mirror dew-point hygrometer, $\Delta T_{d/f\,inhom}$ is the correction due to dew/frost-point temperature inhomogeneity, $\Delta T_{d/f\ cal}$ is the correction of the chilled mirror dew-point hygrometer due to its calibration, $\Delta T_{d/f\ res}$ is the correction due to the resolution of the chilled mirror dew-point hygrometer and $\Delta T_{d/f\ dtb}$ is the correction due to dew/frost-temperature difference between the two branches. In equation (3.7) the values of ΔT_{inhom}, ΔT_{res} and $\Delta T_{d/f\ dtb}$ are assumed to be zero.

3.4.2 Measurement uncertainty

In this work ISO GUM method [20, 21, 23] is used for evaluation of measurement uncertainty. Main steps of this method can be written as follows,

(i) Definition of the measurand
(ii) Establishment of measurement model
(iii) Identification of important uncertainty sources
(iv) Modification of the model
(v) Evaluation of the input quantities and calculation of the value of the measurand
(vi) Estimation of standard uncertainties of input quantities
(vii) Calculation of combined standard uncertainty of the measurand
(viii) Calculation of expanded uncertainty
(ix) Examine the uncertainty budget

The definition of the measurand is the first step of uncertainty evaluation and in this work, measurand is the relative humidity correction of the hygrometer under calibration or that of reference hygrometers. Equation (3.1) can be considered as the basic measurement model [20-22, 25, 26].

For the calculation of combined standard uncertainty the law of propagation of uncertainties [20-22] is used. After applying this law to the equation (3.1) it can be written as follows,

$$u^2(RH_{corr}) = u^2(RH_{ref}) + u^2(RH_{mea}) \tag{3.8}$$

Equation (3.8) can be used for uncertainty evaluation of reference hygrometer calibration as well as for external hygrometer calibration.

In case of reference hygrometer calibration $u(RH_{ref})$ is the combined standard uncertainty of reference relative humidity value (RH_{ref}) obtained from the chilled mirror dew-point hygrometer readings and calibrated temperature sensors readings. $u(RH_{mea})$ is the combined standard uncertainty of relative humidity value (RH_{mea}) due to the hygrometers under calibration.

$u\left(RH_{ref}\right)$ can be found by the following formula:

$$u\left(RH_{ref}\right) = RH_{ref} \cdot \sqrt{\left(\frac{u(E_w(T_d))}{E_w(T_d)}\right)^2 + \left(\frac{u(E_w(T))}{E_s(T)}\right)^2} \quad , \tag{3.9}$$

According to equations (3.3 and 3.9), it is necessary to find the combined standard uncertainties of the saturated water vapor pressure $E_w(T)$ at temperature T and saturated water vapor pressure at dew-point temperature $E_w(T_d)$ to find the combined standard uncertainty of the relative humidity reference $u\left(RH_{ref}\right)$ value.

Uncertainty evaluation of saturated water vapor pressure $E_w(T)$ at temperature T can be carried out according to the law of propagation of uncertainties applied to equation (3.4):

$$u^2(E_w(T)) = \left(\frac{\partial E_w(T)}{\partial T}\right)^2 u^2(T) \tag{3.10}$$

Where the partial derivative of saturated water vapor pressure with respect to temperature is calculated by the following equation that is derived from equation (3.4).

$$\frac{\partial E_w(T)}{\partial T} = \ e^{(A\,T^{-1} + \,B + CT + DT^2 + E\ln T)}.\left(-AT^{-2} + C + 2DT + E\left(\frac{1}{T}\right)\right) \tag{3.11}$$

Combined standard uncertainty of air temperature T in the measurement vessel $\left(u(T)\right)$ is evaluated using the following equation:

$$u(T) = \sqrt{u^2(T_{ins}) + u^2(T_{inhom}) + u^2(T_{cal}) + u^2(T_{res})} \tag{3.12}$$

The uncertainty components of temperature instability $u(T_{ins})$ and inhomogeneity in the measurement vessel $u(T_{inhom})$ as well as uncertainties due to the resolution of temperature sensor $u(T_{res})$ and uncertainty due to calibration of temperature sensors $u(T_{cal})$ are considered important.

Combined standard uncertainty of saturated water vapor pressure at dew/frost-point temperature $E_s(T_{d/f})$ is calculated in a similar way to equations (3.10 and 3.11).

Combined standard uncertainty of dew-point temperature $\left(u(T_d)\right)$ is calculated from the following equation:

$$u(T_{d/f}) = \sqrt{u^2(T_{d/f\ ins}) + u^2(T_{d/f\ inhom}) + u^2(T_{d/f\ cal}) + u^2(T_{d/f\ res}) + u^2(T_{d/f\ dtb})} \quad (3.13)$$

In this case uncertainty components of dew-point temperature instability $u(T_{d/f\ ins})$, inhomogeneity $u(T_{d/f\ inhom})$ in the measurement vessel as well as uncertainties due to the resolution $u(T_{d/f\ res})$ and calibration $u(T_{d/f\ cal})$ of the chilled mirror dew-point hygrometer have been taken into account. In addition to that uncertainty component due to the dew/frost-point temperature differences between the two branches of measurement vessel and chilled mirror dew-point hygrometer $u(T_{d/f\ dtb})$ are considered as important.

Standard uncertainty due to reproducibility of corrections of relative humidity $u(\Delta R)$ has also identified as an important uncertainty component. In this case main mathematical model (equation (3.1)) has to be modified as follows in order to include the uncertainty component due to reproducibility of corrections of relative humidity $u(\Delta R)$,

$$RH_{corr} = RH_{ref} - RH_{mea} + \Delta R, \quad (3.14)$$

where $\Delta R = 0$

Then equation (3.8) is slightly changed to the following equation:

$$u^2(RH_{corr}) = u^2(RH_{ref}) + u^2(RH_{mea}) + u^2(\Delta R) \quad (3.15)$$

Equations (3.14) and (3.15) can be used as the final mathematical models for relative humidity correction and uncertainty evaluation.

For the external hygrometer calibration the standard uncertainty associated with the reference value of relative humidity $u(RH_{ref})$ is the uncertainty evaluated previously (calibration of reference hygrometer).

In both cases $u(RH_{mea})$ can be obtained from the standard uncertainties due to resolution $u(res)$ and repeatability $u(rep)$ of the hygrometer under calibration.

Instability of air temperature and dew-point temperature uncertainties belongs to type A category. Standard uncertainties due to instability of air temperature $u(T_{ins})$ and dew/frost-point temperature $u\left(T_{d/f\ ins}\right)$ are evaluated by the highest standard deviations of the three sensors' temperature readings and chilled mirror dew-point hygrometer readings, respectively.

Standard uncertainty due to air temperature inhomogeneity $u(T_{inhom})$ in the measurement vessel can be evaluated using maximum difference between the average temperature and individual temperatures of the three sensors. Standard uncertainty due to dew/frost-point temperature inhomogeneity $u\left(T_{d/f\ inhom}\right)$ can be evaluated by measured dew/frost-point temperature differences between the two measurement locations using probe swapping. Here air temperature and dew/frost-point temperature inhomogeneity corrections are considered to be distributed rectangularly.

Calibration uncertainty of the chilled mirror dew-point hygrometer $u\left(T_{d/f\ cal}\right)$ can be taken from its calibration certificate and for the temperature sensors uncertainty $u(T_{cal})$ has been evaluated in this study (Appendix 1). These two uncertainty components are considered as normally distributed.

Resolution uncertainty of the chilled mirror dew-point hygrometer $u\left(T_{d/f\ res}\right)$ and the temperature sensors $u(T_{res})$ can be obtained using the smallest increments of readings of the instruments.

Uncertainty due to the dew/frost-point temperature difference between the chilled mirror dew-point hygrometer and the measurement vessel branches $u\left(T_{d/f\ dtb}\right)$ can be estimated using the results of the performance tests (section 3.3.3). Uncertainties due to inhomogeneity, calibration, dew/frost-point temperature differences and resolution belong to type B category. Standard uncertainty due to reproducibility of relative humidity corrections $u(\Delta R)$ can be evaluated as pooled standard deviation. Gum Workbench software was used for uncertainty evaluation.

Finally, expanded uncertainty can be found by the following equation:

$$U(RH) = k \cdot u(RH) \tag{3.16}$$

where *k* is the suitable coverage factor [20-22, 27].

4. Results and Discussion

4.1 Performance tests

4.1.1 Dew/frost-point temperature stability and homogeneity tests.

As discussed in section 3.3.1 stability and homogeneity were checked for compressed, moist and mixed air separately. The main idea of checking homogeneity and stability was to estimate the corresponding uncertainty components.

The compressed air stability was examined in terms of frost-point temperature stability in the measurement vessel.

Figure 5 (a) shows the frost-point temperature temporal decrease in the measurement vessel for compressed air and figure 5 (b) shows the frost-point temperature temporal variation after 900 minutes of stabilization.

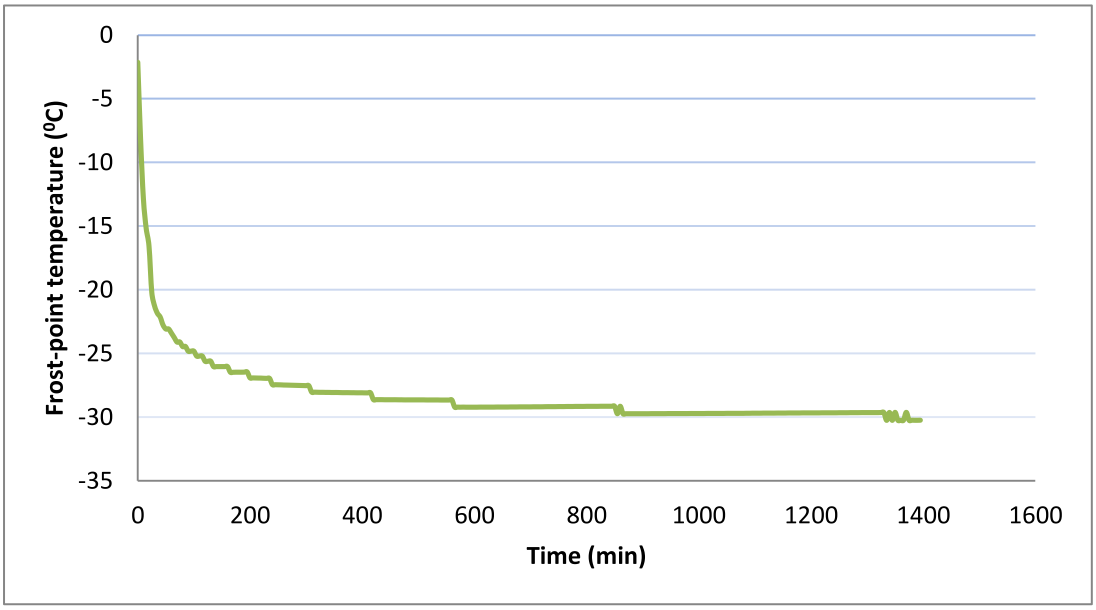

Figure 5(a). Frost-point temperature temporal decrease in the measurement vessel for compressed air.

According to figure 5(a) it can be noticed that at the beginning frost point temperature was just below 0 °C and it was continuously dropping. After 900 minutes (approximately 15 hours) frost point temperature in the measurement vessel was almost stabilized. Stability of compressed air can be achieved by running the established system overnight. Stabilization time could be reduced if smaller volume vessel is used instead of the existing one that was chosen for convenient calibration of different types of external hygrometers.

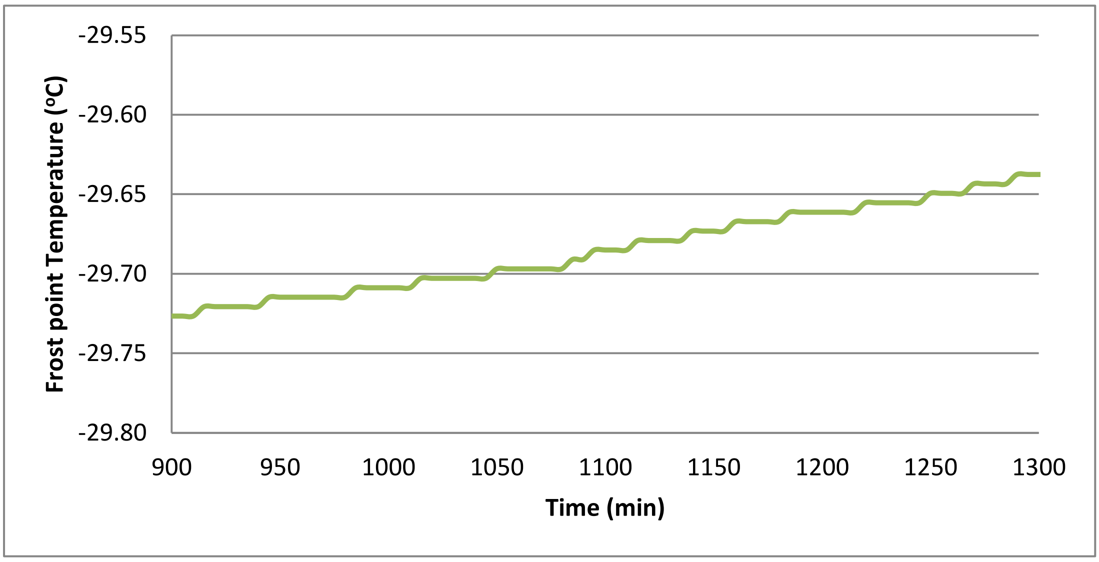

Figure 5(b). Frost-point temperature temporal variation in the measurement vessel for compressed air after 900 minutes of stabilization.

According to figure 5(b) it is clear that during the time interval from 900 minutes to 1300 minutes frost-point temperature of compressed air changed by 0.1 ^{o}C. But during the calibration in one calibration point readings were taken within short period of time (20 minutes). During that time period the frost point temperature change was significantly less than 0.1 ^{o}C.

For the moist and mixed air stability was evaluated similar to the compressed air case.

Figure 6 shows the dew-point temperature temporal change in the measurement vessel for moist air.

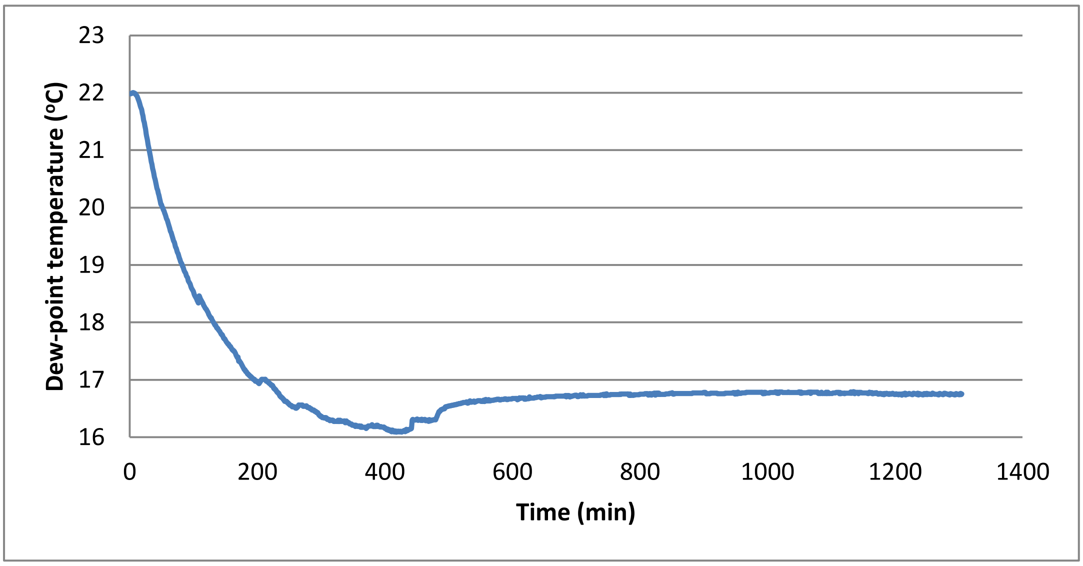

Figure 6. Dew-point temperature temporal change in the measurement vessel for moist air.

It can be noticed that after 700 minutes dew-point temperature was stable in the measurement vessel.

Mixed air dew/frost-point temperature instability was checked for different relative humidity values as 2%, 5%, 10%, 15% and 30%.

Figure 7 shows the dew/frost-point temperature variation in the measurement vessel for different humidity levels.

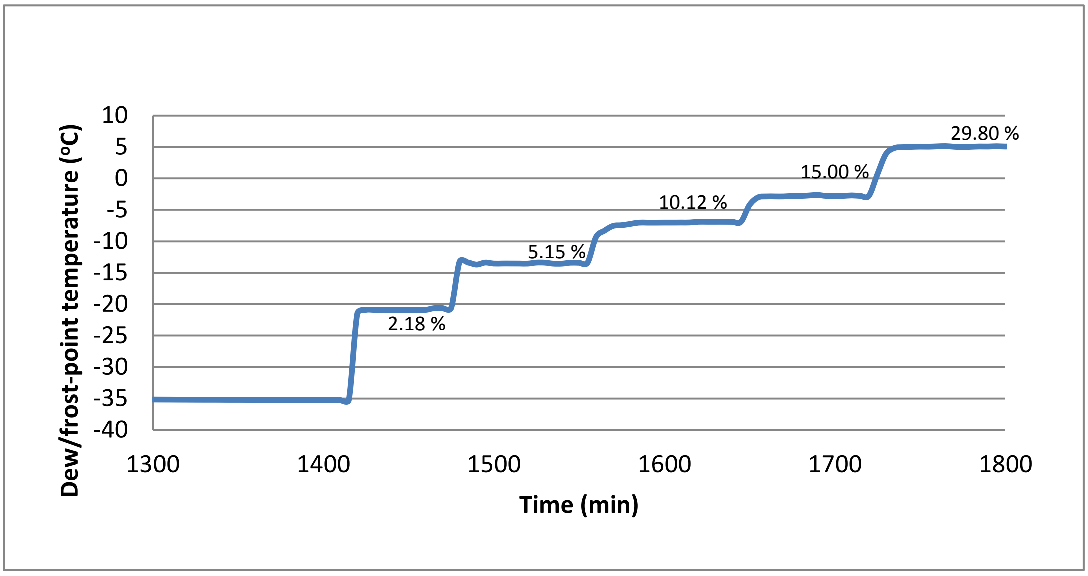

Figure 7. Dew/frost-point temperature variation in the measurement vessel for mixed air at air temperature range 23.0(±0.3) ^{o}C. The horizontal regions where dew/frost-point temperatures are stable were marked with corresponding relative humidity values.

Standard uncertainties due to inhomogeneity were evaluated for mixed air. Table 3 shows the homogeneity test results for 2% and 30% relative humidity levels.

Table 3. Inhomogeneity of mixed air.

Air type	Standard uncertainty from the test results (K)	Estimated standard uncertainty (K)
Mixed air (2%)	0.02	0.04
Mixed air (30%)	0.01	0.04

4.1.2 Air temperature stability and homogeneity in the measurement vessel.

Since air temperature is an important factor in humidity measurements and calibration of hygrometers, it was evaluated using mixed air. In other words air temperature inside the measurement vessel was measured with different relative humidity levels (2%, 5%, 10%, 15% and 30%). All measurements were carried out according to the procedure written in section 3.3.2. All recorded temperature measurements from three sensors were corrected according to the calibration details.

Figure 8 shows the typical temperature stability in the measurement vessel during the whole measurement process within roughly 29 hours.

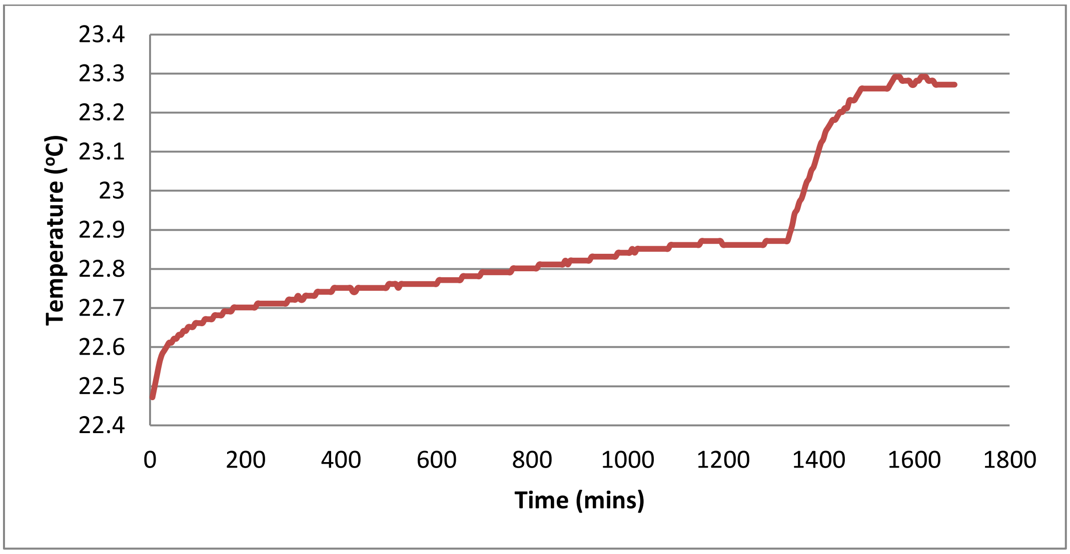

Figure 8. Temperature stability in the measurement vessel during the whole process.

It was noticed that during the whole process (system stabilization + calibration) air temperature maximum variation was about 0.8 ^{o}C.

Calibration process was carried out after the 1300 minutes of stabilization and it was noticed that during the calibration process variation of air temperature inside the vessel was about 0.4 ^{o}C. But during the calibration in one calibration point readings were taken within short time period (20 minutes). During this short period the air temperature change was significantly less than 0.4 ^{o}C.

Homogeneity of air temperature was evaluated using three temperature sensors. Figure 9 characterizes the temperature homogeneity in the measurement vessel during the whole process.

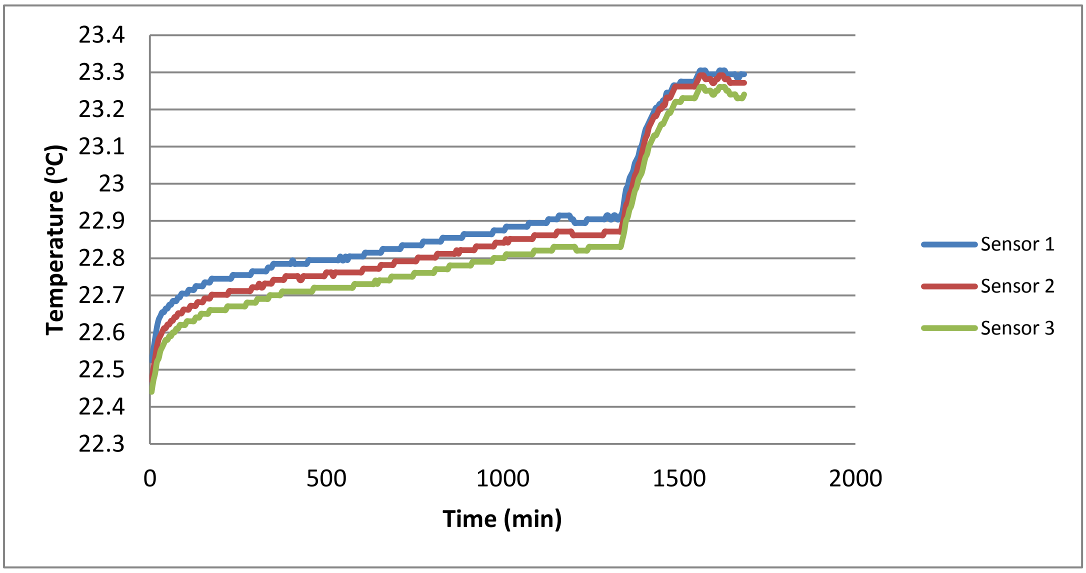

Figure 9. Temperature homogeneity in the measurement vessel during the whole process can be found as the maximum air temperature difference between the corrected readings of the three thermometers.

It was noticed that the readings of the three sensors agreed relatively well. Maximum air temperature deviation between the three thermometers was less than 0.1 ^{o}C.

4.1.3 Test results of comparison of humidity values between the measurement vessel and the chilled mirror dew-point hygrometer branches.

Standard uncertainties due to dew/frost-point temperature difference between the chilled mirror dew-point hygrometer and the measurement vessel branches were evaluated for mixed air. Table 4 shows the corresponding test results for 2% and 30% relative humidity levels.

Table 4. Test results of comparison of dew/frost-point temperature values between the measurement vessel and the chilled mirror dew-point hygrometer branches.

Air type	Standard uncertainty from the test results (K)	Estimated standard uncertainty (K)
Mixed air (2%)	0.10	0.2
Mixed air (30%)	0.15	0.2

4.2.1 Uncertainty budget for reference hygrometer.

Table 5 and 6 shows the uncertainty budgets for 2% and 30 % relative humidity levels. These uncertainty budgets include all input quantities, their values, probability density functions, standard uncertainties, sensitivity coefficients and their contributions.

Table 5. Uncertainty budget for one sensor at 2% relative humidity level.

Quantity	Value (K)	Probability density function	Standard uncertainty u_i(K)	Sensitivity coefficient c_i(%·K^{-1})	(C_i*u_i) (%)	Contri-btion (%)
$T(average)$	296.2808	Normal	0.0118	-0.14	$-1.6\cdot10^{-3}$	0.0
ΔT_{inho}	0.0	Rectangular	0.0289	-0.14	$-3.9\cdot10^{-3}$	0.3
ΔT_{res}	0.0	Rectangular	$2.89\cdot10^{-3}$	-0.14	$-390\cdot10^{-6}$	0.0
ΔT_{cal}	-0.055	Normal	0.0150	-0.14	$-2.0\cdot10^{-3}$	0.0
$T_{d/f}(average)$	-25.2455	Normal	0.0207	0.23	$4.7\cdot10^{-3}$	0.4
$\Delta T_{d/f\ inho}$	0.0	Rectangular	0.0231	0.23	$5.2\cdot10^{-3}$	0.5
$\Delta T_{d/f\ res}$	0.0	Rectangular	$2.89\cdot10^{-3}$	0.23	$650\cdot10^{-6}$	0.0
$\Delta T_{d\ cal}$	0.0	Normal	0.0650	0.23	0.015	4.1
$T_{d/f\ dtb}$	0.0	Rectangular	0.0115	0.23	0.026	13.0
RH_{rep}	3.69	Normal	0.0100	-1	-0.010	1.9
RH_{res}	0.0	Rectangular	0.0289	-1	-0.029	15.9
ΔR	0.0	Rectangular	0.0577	1	0.058	63.7
RH_{corr}	-1.44%					
$U(RH_{corr})$	0.14%					

Table 6. Uncertainty budget for one sensor at 30% relative humidity level.

Quantity	Value (K)	Probability density function	Standard uncertainty u_i (K)	Sensitivity coefficient c_i (%·K^{-1})	(C_i*u_i) (%)	Contri-btion (%)
$T(average)$	296.3977	Normal	$4.35 \cdot 10^{-3}$	-1.8	- $7.8 \cdot 10^{-3}$	0.0
ΔT_{inho}	0	Rectangular	0.0289	-1.8	- 0.052	2.8
ΔT_{res}	0	Rectangular	$2.89 \cdot 10^{-3}$	-1.8	- $5.2 \cdot 10^{-3}$	0.0
ΔT_{cal}	-0.055	Normal	0.015	-1.8	-0.027	0.8
$T_{d/f}(average)$	4.5915	Normal	$5.82 \cdot 10^{-3}$	2.1	0.012	0.2
$\Delta T_{d/f\ inho}$	0	Rectangular	0.0231	2.1	0.048	2.4
$\Delta T_{d/f\ res}$	0	Rectangular	$2.89 \cdot 10^{-3}$	2.1	$6 \cdot 10^{-3}$	0.0
$\Delta T_{d\ cal}$	0	Normal	0.065	2.1	0.14	19.0
$\mathrm{T}_{\mathrm{d/f\ dtb}}$	0	Rectangular	0.115	2.1	0.24	59.8
RH_{rep}	29.48	Normal	0.020	-1	-0.02	0.4
RH_{res}	0	Rectangular	0.0289	-1	-0.029	0.9
ΔR	0	Rectangular	0.115	1	0.12	13.7
RH_{corr}	0.45%					
$U(RH_{corr})$	0.62%					

At 2% relative humidity level the dominant uncertainty source is reproducibility of the relative humidity corrections and its contribution is 63.7%. The uncertainty components due resolution of hygrometer under calibration (reference hygrometer) and the dew/frost-point temperature difference between the chilled mirror dew-point hygrometer and the measurement vessel branches contribute 15.9% and 13%, respectively. At 30% relative humidity level uncertainty due to the dew/frost-point temperature difference between the chilled mirror dew-point hygrometer and the measurement vessel plays the dominant role and its contribution is 59.8% and uncertainty due to calibration of chilled mirror dew-point hygrometer and reproducibility of the relative humidity corrections contributes 19% and 13.7%, respectively.

5. Summary

Two flow system was established in this study for calibrating hygrometers in the low relative humidity levels (2%-30%) at room temperature because it is not possible to generate low humidities in the present climatic chamber. During this work the entire system has been set up for mixing compressed air and moist air to generate different relative humidity values in the measurement vessel. Several tests were carried out for studying the system. Firstly, the dew/frost-point temperature stability of the mixed air was studied. Dew/frost-point temperature homogeneity was studied for the 2% and 30 % relative humidity levels in the measurement vessel. Additionally air temperature homogeneity and stability were studied in the measurement vessel. Also the comparison of dew/frost-point temperature values between the measurement vessel and the chilled mirror dew-point hygrometer branches was carried out. Temperature sensors of the reference hygrometers were calibrated in water in order to be able to measure air temperature in the measurement vessel accurately enough. The relative humidity sensors of the reference hygrometers were calibrated using the calibrated air temperature sensors and the chilled mirror dew-point hygrometer. The expanded uncertainties (k=2) of reference hygrometers have been estimated to be in the range of 0.2% to 0.7% for the relative humidity range of 2% to 30%.

An external hygrometer can be calibrated using the previously calibrated relative humidity reference hygrometers. It was found that the stabilization time for selected relative humidity values was about 1 hour after the system had been run overnight with compressed air. For relative humidity sensors of high response time the stabilization time can be longer.

Based on the current thesis it can be concluded that it is possible to establish a low cost two-flow system for calibrating hygrometers in the low relative humidity range at room temperature. This system is good enough if time limit for calibrating hygrometers is not very crucial.

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7. Summary in Estonian

Kahe voolu süsteem hügromeetrite kalibreerimiseks toatemperatuuril

Käesolevas töös seati üles kahe voolu süsteem hügromeetrite kalibreerimiseks toa temperatuuril madalatel suhtelise niiskuse väärtustel (2…30) %, kuna madalate niiskuste tekitamine olemasolevas kliimakambris pole realiseeritud. Käesolevas töös seati üles süsteem, mille abil saab segada kuiva suruõhku ja niisket õhku, et tekitada mõõteanumas erinevaid suhtelise niiskuse väärtuseid. Süsteemi uurimiseks viidi läbi mitmeid katseid. Esmalt uuriti segatud õhu kaste- ja härmapunkti temperatuuride stabiilsust. Kaste- ja härmapunkti temperatuuride homogeensust mõõteanumas uuriti suhtelise niiskuse väärtustel vahemikus 2 % ja 30 %. Lisaks uuriti mõõteanumas ka õhu temperatuuri stabiilsust ja homogeensust. Samuti võrreldi kaste- ja härmapunkti temperatuuride väärtuseid mõõteanuma ja kastepunkti peegelhügromeetri õhuvoolu harude vahel. Referentshügromeetrite temperatuuri sensoreid kalibreeriti vees selleks et olla suutelised mõõtma õhu temperatuuri mõõteanumas piisava täpsusega. Referentshügromeetrite suhtelise niiskuse sensoreid kalibreeriti, kasutades varem kalibreeritud temperatuuri sensoreid ja kastepunkti peegelhügromeetrit. Referents-hügromeetrite kalibreerimise laiendmääramatuseks (k=2) hinnati (0,2…0,7) % õhu suhtelise niiskuse vahemikus (2…30) %.

Täiendavat hügromeetrit saab kalibreerida kasutades eelnevalt kalibreeritud referents-hügromeetreid. Magistritöös leiti, et suhtelise niiskuse stabiliseerumiseks mõõteanumas kulub umbes üks tund pärast seda, kui süsteemist on pikema perioodi vältel läbi puhutud kuiva suruõhku. Suure ajateguriga hügromeetrite korral võib stabiliseerumiseks rohkem aega kuluda.

Käesoleva magistritöö põhjal võib järeldada, et on võimalik odavalt realiseerida kahe voolu süsteemi hügromeetrite kalibreerimiseks toa temperatuuril madalatel suhtelise niiskuse väärtustel. Antud süsteem on piisavalt hea, kui hügromeetrite kalibreerimisele kuluv aeg ei ole väga oluline.

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Foremost, I would like to express my sincere gratitude to my supervisor Ph.D Martin Vilbaste for the continuous support of my master thesis research work, for his patience, motivation, enthusiasm, and immense knowledge. His guidance helped me in all the time of research and writing of this thesis.

Besides my supervisor, I would like to thank Viljar Pihl and Eino Rätsep for the help they offered to do some metal work during the system establishment.

9. Appendices

Appendix 1

Results of temperature sensors' calibration.

Sensor No	Serial No	Correction (^{0}C)	Expanded uncertainty(^{o}C),k=2
1	08020160	-0.055	0.022
2	04020125	0.14	0.022
3	03120988	-0.080	0.024

Appendix 2

Results of external hygrometer calibration

RH level (%)	Correction (%)	Expanded uncertainty (%)k=2
2	-2.82	0.16
30	-1.23	0.62

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