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KazStandard Primary Dew/Frost Point Generator

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Abstract: This article presents studies on the dew point/frost temperature generator using the single-pressure (1-P) and single-temperature (1-T) method first developed by KazStandard to improve the calibration accuracy of dew point hygrometers. The method of applying the dew point temperature scale is based on the supply of humid air with high instability through a thermostatic saturator, which enables increased measurement accuracy. The characteristics include a study of the saturator's efficiency, the stability of the dew point temperature reproduction and a comparison with a calibrated dew point hygrometer. The study was conducted in the dew point temperature range from –50°C to 20°C, and the sources of measurement uncertainty were analyzed.

Keywords: standards, measurements, humidity generator, dew point temperature, uncertainty, saturator

1. Introduction

With the ever-changing climate and political instability, any contribution to preventing climate threats that threaten humanity is important, especially when it comes to contributions from developing countries. The control of energy parameters of various media begins with the creation and development of an appropriate quality infrastructure, which includes the development of and compliance with international standards and the metrological traceability of measured and controlled quantities.

Humidity is of great importance in understanding the nature of climate change. However, it is also one of the most important factors affecting product quality. Since humidity varies over a wide range, devices and sensors are needed that can measure it over the entire range of possible changes and ensure traceability.

Traceability of humidity measurements in units of measurement is one of the main components of the quality infrastructure. The main tasks are the measurement of humidity in different units of measurement, such as relative humidity (%), dew /frost point temperature (°C), volume fraction of moisture (ppm) and the transfer of units of measurement to measuring instruments. Metrological characteristics such as uncertainty and measuring range as well as an immersion or flow measurement method play a crucial role in choosing the best type of humidity sensor.

The main activity of the Kazakhstan Institute of Standardization and Metrology (KazStandard) is to ensure the metrological traceability of measurement units within the country, i.e., by calibrating reference instruments of calibration laboratories. In the field of humidity measurement, KazStandard already has a secondary calibration system for dew /frost point calibration, which is used as the state standard in the range of -80°C to 20°C with an uncertainty of ± 0.2 °C to ± 0.8 °C. However, commercial calibration laboratories in the country have different instruments that provide the same level of accuracy as the state standard KazStandard. Therefore, the hierarchy of traceability of measurement units is not followed by the state standard in the country. At the same time, the traceability and repeatability of measurements of calibration device data in certified laboratories of national metrological institutes (NMI) of foreign states is ensured [1].

In addition, it is worth noting that all NMIs of the countries of the Central Asian region also do not have primary dew point standards and are forced to calibrate their working dew point standards in the NMIs of foreign states.

A reference humidity meter is a set of measuring instruments designed to reproduce, store and transmit a humidity unit to other measuring devices. The reproducibility of certified moisture units is based on measurement methods

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defined by standards derived from basic physical principles. Non-certified humidity units then differ in the reproducibility of the measurements.

The fundamental primary humidity standard is a gravimetric hygrometer, which is based on the determination of humidity r (mass ratio) by the mass of wet and dry gas. The water mass values are determined by the direct method, and the dry air mass is determined by the calculation method, in which the density and volume of dry air are determined. The main disadvantages of the gravimetric hygrometer are the duration of the humidity measurement, the limitations in the design of the water absorption system and the resulting influence on the measuring range [2].

A single-pressure humidity generator saturates the gas with a constant gas flow rate at a certain pressure and temperature and works mainly with one or two liquid thermostats to which the pre-saturator and the main saturator are attached. The humidity value td (dew point temperature) is equal to the temperature of the saturator, where the temperature of the saturator is equal to the temperature of the thermostat. The main source of uncertainty is the effectiveness of the saturator [3]-[6].

Two-temperature humidity generators (2-T) are an extension of the single-pressure humidity generator (1-P) with the connection of a relative humidity chamber located in a thermostatically controlled liquid thermostat or climatic chamber. The relative humidity (RH) value is determined in the relative humidity chamber by measuring the temperature values inside the chamber and the known dew point temperature of the supplied 1-P generator. The pressure in the saturator and in the chamber is the same. The main source of uncertainty is the instability of the wet flow in the chamber [7].

Humidity generators of the two-pressure type (2-P) are based on the principle of two pressures, whereby the humidity values RH are determined from the measured pressure values in the saturator and in the chamber, assuming that the temperature in the saturator and in the chamber is the same. The main problem of the saturator is the formation of condensate in the expansion part of the structure and the maintenance of a constant gas flow [8]-[9].

Hybrid type humidity generators are based on the principle of generating humidity with two pressures and a split flow in a single design. In the two-pressure method, the gas is saturated at a higher pressure and then expanded to a lower pressure. In the split flow method, which the generator uses, the saturated gas is diluted with dry gas and the gas flow is accurately measured. With this method, arbitrarily low humidity values can be achieved if the saturator is operated at favorable temperatures [10]-[11].

The most common humidity generator is 1-P, which is explained by the simplicity of implementation and the lesser importance of considering the pressure value, as shown in the EUROMET.T- K6 report, in which 12 out of 24 NMIs use an implementation of this method [12].

The problem of how to ensure the traceability of the dew point temperature unit without degrading the metrological characteristics of the working standards used in commercial calibration laboratories was solved by developing the primary humidity generator based on the available material and technical sources of the thermophysical and temperature measurement laboratory. At the same time, we maintain the state of KazStandard as an NMI. The goal is to further develop the primary humidity generator and make it a KazStandard and be the first in Central Asia.

The following tasks were therefore set as part of the creation of this standard:

- development of a computer model of the saturator and the physical processes within it;
- development of a saturator for the preparation of wet gas with stable output characteristics;
- direct measurement of the dew /frost point temperature with a thermometer calibrated according to the state standard of temperature and whose metrological characteristics are confirmed by additional comparisons [13];
- estimation of the uncertainty of the developed humidity generator and its comparison with the current dew point measurement standard.

2. Subject & Methods

A. Single-pressure dew point generator

The main components of the generator are an oil-free air compressor with a receiver, a dehumidifier, a dew point generator, a liquid thermostat, a heat exchanger, a saturator, two standard platinum resistance thermometers (SPRT), an AC resistance bridge thermometer, and a chilled mirror hygrometer (CMH).

The operating principle of a humidity generator with one pressure of 1-P and one temperature (1-T) is to humidify the gas at a certain temperature, where the temperature of the bath is equal to the dew point temperature, and at a constant pressure of the humidified gas flow. Fig. 1 shows the diagram of the KazStandard humidity generator.

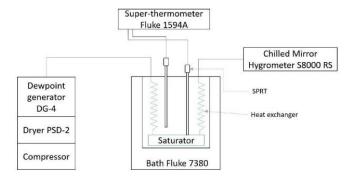


Fig. 1. Diagram of the KazStandard humidity generator.

The almost completely humidified air is prepared in the DG-4 dew point generator (Michell Instruments), which includes a PSD-2 dehumidifier and an oil-free air compressor with a 10-liter receiver. The stability of temperature maintenance by the DG-4 dew point generator is estimated to be $\pm 0.5\,^{\circ}$ C. The humidified air from the DG-4 dew point generator enters an external heat exchanger. The air flow is regulated by a needle valve located in the generator itself. The flow rate is not greater than 1 l/min. The heat exchangers are made of stainless steel, the diameter of the inner tube is 4 mm, that of the outer tube is 6 mm, the total length of the tube is

2.95 m. They are located together with the saturator in a Fluke 7380 liquid thermostat with a bath volume of 4 liters. The humidified air entering the external heat exchanger is completely cooled/heated to the temperature of the thermostat and then enters the saturator. In the saturator, the air flows over the surface of the water/ice, ensuring complete saturation. The saturator is a horizontally arranged oval vessel with a width of 15 mm, a height of 30 mm and a length of 200 mm to ensure a long passage of the air flow over the surface of the water / ice, which is almost half filled with water/ ice. The diagram of the saturator with heat exchangers is shown in Fig. 2. When the temperature of the thermostat stabilizes, it is equal to the temperature of the saturator Ts, and the temperature of the saturator is equal to the dew point temperature Td. At the outlet of the air from the saturator, one SPRT is installed to measure the temperature at the outlet of the saturator, and the other SPRT is immersed in a liquid thermostat to measure the temperature of the bath. After the air leaves the saturator, it flows through an internal heat exchanger for additional temperature stabilization and then enters a portable reference hygrometer.

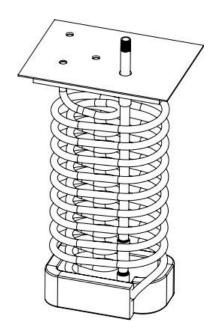


Fig. 2. Drawing of a saturator with a heat exchanger.

B. Method

The implementation of the reference unit for measuring the dew point temperature was carried out as follows:

- to obtain a positive the dew point temperature, the DG-4 dew point generator brings air into the saturator, which is set to a temperature of −20 °C to −10 °C;
- at a negative dew point temperature, the DG-4 dew point generator supplies air to the saturator at a temperature that is 5 °C higher than the temperature in the liquid thermostat.

The transition to a stable measurement mode at a negative dew point temperature was 4 hours, at a positive dew point temperature -20-30 minutes. The graph below shows the transition to measurement mode at a dew point temperature of -18 °C (Fig. 3).

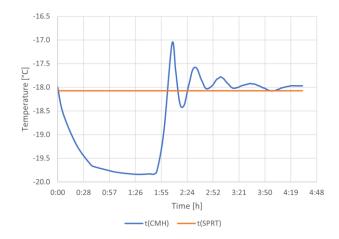


Fig. 3. Switching to negative dew point temperature of −18 °C.

C. Portable reference hygrometer

For comparison, a hygrometer with chilled mirror «S8000 RS» from Michell Instruments was used, which operates in the dew point temperature range of -90 °C to 20 °C. Traceability is guaranteed by the National Physical Laboratory (NPL), UK.

3. RESULTS & DISCUSSION

During the development of the saturator, computer models were initially built using the COMSOL Multiphysics program in order to investigate the thermodynamic and hydrodynamic processes at the saturator. During the simulation, the approximate time to reach a stable regime, the temperature difference between the bath and the saturator, the temperature distribution in the saturator and the saturation efficiency were determined, which are shown in Fig. 4 and Fig. 5.

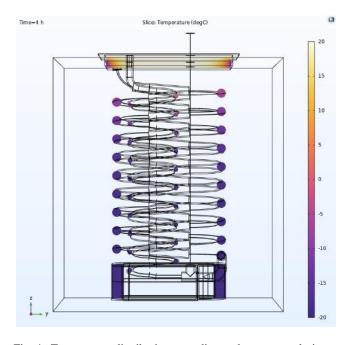


Fig. 4. Temperature distribution according to the saturator design at a bath temperature of -20 °C.

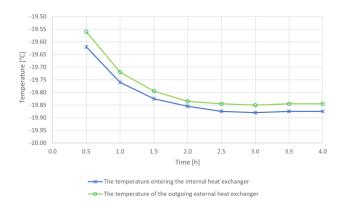


Fig. 5. Dew point temperature stabilization.

The obtained experimental measurement results of the 1-P/1-T generator are shown in the dew point temperature range from -50 °C to 20 °C, increasing by 10 °C (i.e., -50, -40, -30, -20, -10, 0, 10, 20 °C). Table 1 shows the measurement results. The results are the average values over four cycles for each set point, and the readings were taken within 20 minutes.

The overall stability of maintaining the dew point temperature of the generator is ± 0.01 °C. When measuring the temperature of the bath and the saturator at each set point, it was found that the temperature difference is not more than 0.003 °C, which does not significantly affect the measurement results. Thus, the bath temperature value is sufficient to be used as a reference value when calibrating a chilled mirror hygrometer.

The saturator efficiency was determined by the difference between the temperature values of SPRT and CMH. A change in the air flow velocity in the range of 0.5 to 1 l/min has no effect on the efficiency of the saturation system as the generator's performance does not change. Table 2 shows an example of the uncertainty budget calculation at a dew point temperature of $-10~^{\circ}\mathrm{C}$ with an uncertainty of $\pm 0.14~^{\circ}\mathrm{C}$ [6]-[7]. The main source was the effectiveness of the saturator. Thus, in the dew point temperature range considered, the measurement error was found to be between 0.06 and 0.52 $^{\circ}\mathrm{C}$ at a dew point temperature of -50 to 20 $^{\circ}\mathrm{C}$. Fig. 6 shows the difference in uncertainty before and after the development of the humidity generator.

Table 1. Measurement results.

No.	Saturation temperature t(SPRT)	Measured value of the hygrometer t(CMH)	Difference $\Delta t = t(SPRT) - t(CMH)$	Extended uncertainty
	[°C]	[°C]	[°C]	[°C]
1	-50.074	-49.63	-0.444	0.52
2	-40.077	-39.83	-0.247	0.29
3	-30.058	-29.96	-0.098	0.13
4	-20.025	-19.95	-0.075	0.11
5	-10.076	-9.97	-0.106	0.14
6	0.034	0.05	-0.016	0.06
7	10.006	9.92	0.086	0.12
8	20.008	19.91	0.098	0.13

Table 2. Uncertainty budget at −10 °C dew point temperature.

Source	Value	Distribution	Divisor	Sensitivity coefficient	Standard uncertainty
Repeatability SPRT	0.001	Normal	1	1	0.001
SPRT calibration	0.003	Normal	2	1	0.0015
SPRT resolution (indicator unit)	0.001	Rectangular	3.46	1	0.000289
Bridge calibration	0.0001	Normal	2	2.6	0.00013
Bath stability	0.01	Rectangular	1.73	1	0.00578
Bath homogenity	0.01	Rectangular	1.73	1	0.00578
Saturator efficiency	0.106	Rectangular	1.73	1	0.06127
Dew-point hygrometer calibration	0.06	Normal	2	1	0.03
Resolution of hygrometer	0.01	Rectangular	3.46	1	0.00289
Combined uncertainty		0.069			
Expanded uncertainty (k = 2)		0.14			

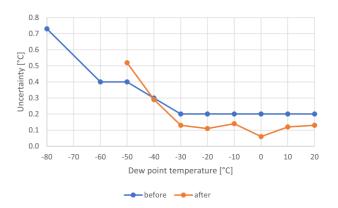


Fig. 6. The difference in uncertainty between before and after the development of a humidity generator.

The effectiveness of the saturator directly depends on the volume of the thermostatic bath and the length of the water/ice flow in the saturator, which affects the results when the maximum deviation reaches 0.5 °C. This study was performed in a small 4-liter bath for which a 90 ml saturator was made with a flow length of 200 mm above the water/ice surface, while other national metrology institutes, such as TUBITAK UME (Turkey), use a saturator in a bath volume of 16 liters and a maximum generator uncertainty of ±0.08 °C (at -60 °C) [5], MIKES (Finland) 22 liters and ± 0.116 °C (at -90 °C) [16], LPM (Croatia) 30 liters and ± 0.074 °C (at -70 °C) [6], MIRS/FE-LMK (Slovenia) 60 liters and ± 0.046 °C (at -50 °C) [14]-[15], MBW Calibration AG (Switzerland) – 27 liters and ± 0.05 °C (at -60 °C) [17], 167 liters and ± 0.008 °C (at -70 °C) [10]. Fig. 7 shows the dependence of the generator's uncertainty on the volume of the saturation bath. The uncertainty decreases with increasing bath volume and with the volume of the saturator.

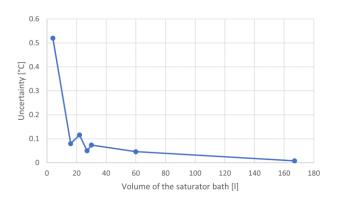


Fig. 7. Dependence of the uncertainty of the generator on the volume of the saturator bath.

The DG-4 dew point generator is based on a flow mixing method that depends on the desiccant PSD-2, which contains 4A molecular sieves that deteriorate over time to keep the air humidity stable and deviate from the installation point. The developed humidity generator 1-P/1-T makes it possible to increase the stability of the supplied humid air, make the dew/frost point temperature setting dependent on the thermostat and improve the reproducibility of the dew/frost point temperature unit.

4. CONCLUSION

KazStandard has developed the first primary humidity standard in Kazakhstan and Central Asia. It is a 1-P/1-T humidity generator with a small volume of a saturation bath that has been successfully tested in the range of -50 °C to 20 °C with an uncertainty of 0.06 °C to 0.52 °C. The effectiveness of the saturator, which was the main source of uncertainty, was also determined.

A computer model of the hydrodynamic and thermodynamic processes in the saturator showed positive results and served as the basis for the development of a physical model of the humidity standard.

An assessment of the stability of the developed saturator showed that its performance to a stable mode of maintaining air humidity lasts 4 hours and does not deviate from ± 0.01 °C, which confirms the high reproducibility of the humidity generator.

With the SPRT thermometer, you can measure the dew point temperature with greater accuracy than with the CMH thermometer, which is installed in accordance with the current state standard for dew point temperature. The advantages of the SPRT thermometer are its portability and compliance with the state standard for temperature.

The value of the saturator efficiency in the range of -30 °C to 20 °C did not exceed 0.1 °C, which enables the calibration of hygrometers with a dew point in this range with an uncertainty of ± 0.14 °C.

Since the developed humidity generator has better characteristics in the range of -30 °C to 20 °C than the current state standard of the dew point temperature unit, it can be used as the primary standard of the dew point temperature unit.

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