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Frost/Dew Point Temperature and Relative Humidity Measurements: Primary and Secondary Calibration Methods

Andreia Furtado [1*], *Fitsum* Zenebe[1], *Özkan* Subas[1], *Michael* Schwinghammer[1], and *Praveen* Giri[1]

[1]Process Insights GmbH, Calibration Center, Max-Eyth-Str. 30, 70736 Fellbach, Germany

Abstract. Humidity measurements are crucial across various industries to ensure product quality, safety, and process efficiency. These measurements can be expressed as absolute quantities, such as frost/dew point, temperatures point, t_f/t_d, or as relative humidity, U, which relates to saturation at a given temperature. Chilled-mirror hygrometers are widely used in high-precision industrial and research applications due to their broad measurement range, stability, and accuracy. Ensuring metrological traceability requires rigorous calibration using traceable methods. The DAkkS-accredited Calibration Laboratory of Process Insights follows DKD-R 5-8 [1] guidelines and meets ISO 17025 [2] requirements, employing both primary and secondary calibration methods for t_f/t_d over the range [-90, 95] °C, and U over the range [1, 98] %rh. Primary calibration achieves the lowest uncertainties using in-house 1-pressure and 2-pressure humidity (U_{t_f/t_f}: 0.4 K - 45 mK and U_U: 0.10 - 0.39 %rh), while secondary calibration relies on stable CM hygrometers and commercial humidity generators (U_{t_f/t_f}: 0.54 K - 70 mK and U_U: 0.10 - 0.49 %rh). For relative humidity calibration, both t_f/t_d [1] and temperature [3] sensors require calibration. Humidity calculations follow established references, ensuring high accuracy [4, 7-8]. This work outlines the methodologies and challenges in maintaining traceability and accuracy in high accuracy humidity measurements at PI Cal.

1 Introduction

Humidity measurement plays a crucial role in various industrial and scientific applications, ensuring product quality, safety, and process efficiency. The characterization of humidity can be divided into absolute and relative quantities. Absolute humidity measurements provide direct information about the amount of water vapor contained in a gas and include parameters such as dew point temperature, t_d and frost point temperature, t_f [4]. On the other hand, relative humidity, U, expresses the relationship between the actual water vapor content and the saturation level at a given temperature [4]. The accurate determination of these parameters is essential across different fields, including climate monitoring, meteorology, pharmaceuticals, semiconductors, and industrial drying processes.

A variety of measurement techniques exist to determine humidity, each differing in complexity, measurement range, and associated uncertainty. Among these, chilled-mirror hygrometers stand out as one of the most reliable and accurate instruments [5], widely used in both industrial and research environments. Their popularity stems from their ability to provide direct, stable, and highly precise measurements over a broad humidity range. However, to maintain confidence in their measurements, metrological traceability is required, necessitating rigorous calibration using traceable methods.

The DAkkS-accredited Calibration Laboratory of Process Insights (PI Cal), GmbH ensures metrological traceability for humidity measurements through a combination of primary and secondary calibration methods. These calibration activities are conducted in compliance with DKD-R 5-8 [1] guidelines and meet the requirements established by ISO 17025 [2].

Frost and dew point temperature calibrations are performed over a range of -90 °C to 95 °C, utilizing both primary and secondary calibration methods. Primary calibration methods achieve the lowest measurement uncertainties by employing single or 1-pressure and 2-pressure humidity generators, which were originally developed by MBW Calibration AG when it functioned as the Swiss Designated Institute for Humidity [6]. These in-house developed generators produce a highly controlled humid gas stream, where the generated humidity is directly traceable to saturation temperature, t_s, and pressure, p_s. In contrast, secondary calibration methods rely on well-characterized chilled-mirror hygrometers with long-term stability, enabling comparative measurements against reference standards. Although secondary calibrations inherently have higher measurement uncertainties, they remain a reliable and widely used method for routine calibration of industrial humidity sensors.

In addition to frost and dew point temperature calibration, relative humidity calibration, in the range of 1 %rh and 98 %rh, is also part of the laboratory's

* Corresponding author: AFurtado@emea.process-insights.com

calibration measurement capabilities (CMC). In the case of chilled mirror hygrometers, this process requires calibration of both dew point/frost point temperature [1] and temperature sensors [3]. To ensure high accuracy across a broad temperature spectrum, humidity calculations follow well-established reference equations and internationally recognized data sources [4,7-8].

This paper aims to describe the operating principles and design of the PI Cal primary humidity generators used for frost/dew point temperature calibration. Additionally, the major uncertainty sources, as well, the acceptance criteria for calibration results, are also presented.

2 Calibration of frost/dew point temperature

2.1 Low range humidity generator (LRG)

2.1.1 Principle of operation

The low range humidity generator (LRG) is a 2-pressure generator based on the saturation of dry air, flowing over a plane surface of isothermal ice at a well-defined temperature, t_s and pressure, p_s.

2.1.2 Design

The LRS consists of 18 stacked electropolished stainless-steel trays, sealed with copper press seals, where ice is formed (Fig. 2). A preceding cooling coil serves as a heat exchanger for the incoming dry air (Fig. 2). Dry air sequentially flows through the trays, repeatedly passing over the ice surface to achieve thermodynamic equilibrium, ensuring full saturation with water at the bath's set temperature. The multiple flow diversions maximize contact with the ice, allowing the air to reach full saturation at the bath's set temperature. The generator operates at a slight overpressure relative to ambient pressure. The measured frost point temperature corresponds to the temperature at which the carrier gas is saturated, with minor corrections applied for pressure drops in the gas flow.

Saturator temperature control is managed via a thermoregulated bath using absolute ethanol as the refrigerant fluid. This setup results in an expanded uncertainty of approximately 2 mK due to instability and 3 mK due to inhomogeneity ($k = 2$).

A constant flow rate of 1 L/min is maintained as the gas mixture is accelerated to sonic velocity through a nozzle, ensuring a stable and controlled flow. Approximately 1.1 to 1.2 L of ultrapure water are required to fill the saturator.

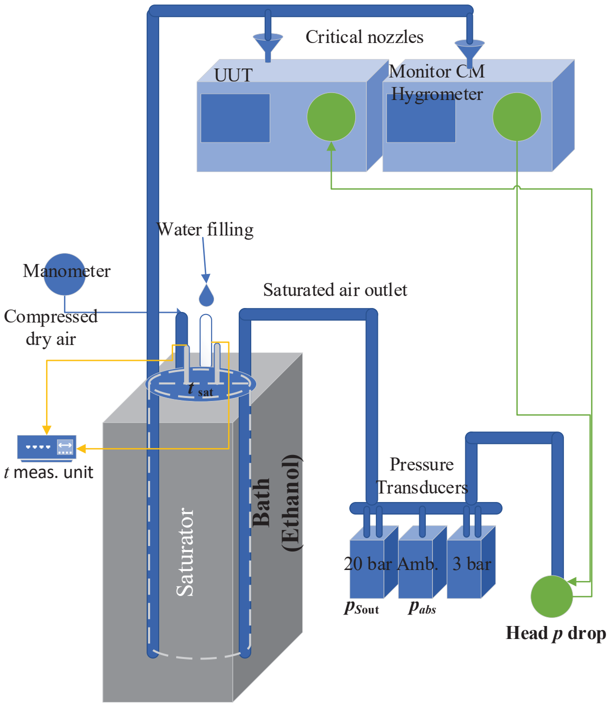

Fig. 1. Schematic diagram of the low range humidity generator (LRS) setup used for the primary calibration of frost/dew point temperature in the interval of [-90, -20] °C.

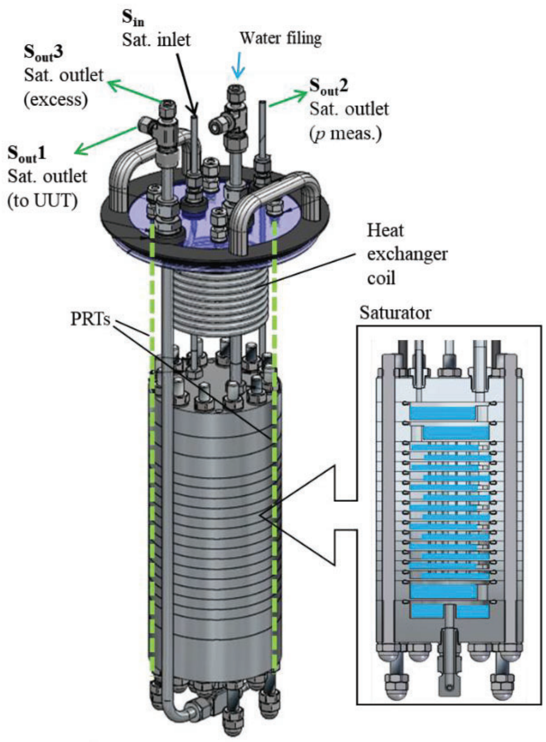

Fig. 2. Schematic diagram of the core of the low range humidity generator (LRS), consisting of a heat exchanger and a saturator. Legend: black arrow – dry air inlet; green arrows – humid air outlet; blue arrow – ultrapure water inlet.

2.2 High range humidity generator (HRG)

2.2.1 Principle of operation

The high-range humidity generator (HRG) operates in either single-pressure mode (1P) or two-pressure mode (2P), depending on the temperature range. In single-pressure mode, it covers dew point temperatures from 1 °C to 95 °C, while in two-pressure mode, it generates frost point temperatures from 0 °C down to - 20 °C.

In single-pressure mode, the dew point temperature corresponds to the temperature at which the carrier gas reaches full saturation. However, corrections must be applied for pressure drops occurring along the gas path to the point of use, including the unit under test and the reference chilled mirror hygrometer(s).

In two-pressure mode, the carrier gas is first saturated at an elevated pressure above ambient conditions. It is then expanded through a controlled expansion valve, leading to a reduction in pressure and temperature, thereby achieving the desired frost point temperature.

2.2.2 Design

The high-range generator (HRG) comprises three main components, all designed and manufactured by MBW [6]: a model G1HX humidity generator, which functions as a precision pre-saturator and pressure controller in two-pressure mode; the HRG main saturator with an integrated heat exchanger; and a motorized expansion valve assembly, connected to the outlet manifold and controlled by the G1HX, for regulating saturator pressure in two-pressure mode (Fig. 3).

Saturator temperature control is achieved using a thermoregulated bath with absolute distilled water as the refrigerant fluid. This configuration results in an expanded uncertainty of approximately 4 mK due to instability and 8 mK due to inhomogeneity ($k = 2$).

Dry air is first introduced into the G1HX pre-saturator, which operates in delta temperature mode at +5 °C relative to the desired frost/dew point temperature. The pre-conditioned humid air is then delivered via a heated stainless-steel hose to the saturator inlet. After achieving full saturation, the humid air exits the saturator through a heated stainless-steel hose, returning to the G1HX manifold before being directed to the unit under test (UUT) and the reference chilled mirror hygrometer. Heated stainless-steel hoses maintain a temperature approximately 30 °C above the frost/dew point temperature to prevent condensation.

The primary heat exchange and condensate formation occur within the heat exchanger coil, where condensation accumulates in the main saturator. As a result, only the final few mK of temperature drop take place inside the main saturator [6] (Fig. 4).

The HRG main saturator has a nominal internal volume of approximately 1 L. While the primary saturation surface is the internal wall of the cylindrical chamber, the equilibrium vapor pressure is primarily defined by the flat surface at the bottom of the bath, which is estimated to be a few mk lower than the wall temperature [6].

The generator operates as close as possible to atmospheric pressure, depending on the pressure resistance and internal flow restrictions of the unit under test. An individual flow rate of 1 L/min per unit under test is used for t_f/t_d below 0 °C, and 0.5 L/min per unit under test is used for t_f/t_d above 0 °C.

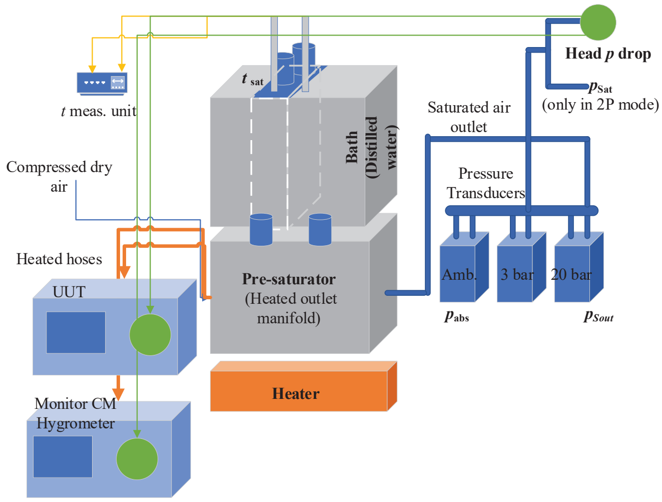

Fig. 3. Schematic diagram of the high range humidity generator setup used for the primary calibration of frost/dew point temperature in the interval of [-20, 95] °C.

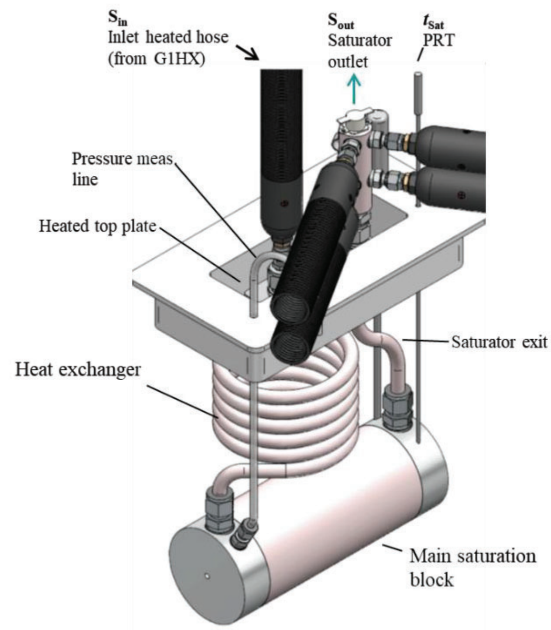

Fig. 4. Schematic diagram of the core of the high range humidity generator, consisting of a heat exchanger and a saturator.

2.3 Dry air

The air is compressed using an oil-free compressor, dried with a molecular sieve heat-regenerator dryer, and then fed through stainless-steel tubing to the saturator inlet. The fully saturated air exits via stainless-steel tubes to the unit under test and reference chilled-mirror hygrometers.

2.4 Temperature and pressure measurement in the saturators

Temperature measurement in the main saturator of each generator is performed using two platinum resistance thermometers (PRTs), calibrated at fixed points, with an expanded measurement uncertainty of 5.5 mK ($k = 2$). In the LRG, both PRTs are immersed in the bath fluid near the saturator exit (Fig. 2). In the HRG, one PRT is placed in the saturator block at the saturator exit, while the other is positioned in the bath fluid at the saturator exit side and serves as a verification sensor (Fig. 4). The PRT are used with resistance measuring units with an expanded measurement uncertainty of 1.2 mΩ ($k = 2$)

Pressure measurements are carried out using three digital pressure gauges in each generator. An absolute pressure transmitter, with an expanded measurement uncertainty of 0.29 hPa ($k = 2$), is used to measure ambient pressure. A 3 MPa differential pressure transmitter, with an expanded measurement uncertainty of 0.29 hPa ($k = 2$), is used to measure the pressure drop at the unit under test and the pressure drop in the LRS saturator. Another 20 MPa differential pressure transmitter, with an expanded measurement uncertainty of 0.79 hPa ($k = 2$), is used to measure the saturator pressure.

In the LRG, the saturator pressure measurement instrument is connected to a purged line with a large diameter. In the HRG, pressure measurement instruments are connected via two-way valves, allowing purging of the measurement lines during changes in saturator temperature and pressure to prevent condensation.

The above-mentioned expanded measurement uncertainties account for uncertainties from calibration, resolution, drift, and hysteresis. For temperature measurements, additional sources of uncertainty include nonlinearity, self-heating, and thermal coupling. For pressure measurements, interpolation, zero error, and height differences are also considered.

3 Calculation Formulas

Ideally, the humid gas exiting the generators (LRG and HRG), with a well-defined saturation temperature, t_{sat} and pressure, p_{sat}, is characterized by a water vapour mole fraction, x_{w}, given by the following equation [7]:

$$x_{\mathrm{w}} = \frac{e_{\mathrm{i,w}}(t_{\mathrm{s}})}{p_s} f_{\mathrm{i,w}}(p_s, t_{\mathrm{s}}) \quad (1)$$

where $e_{\mathrm{i,w}}(t_{\mathrm{s}})$ represents the saturation vapour pressure in the pure phase, with respect to ice (i) in LRG or water (w) in HRG at the temperature t_{s}. The term p_s denotes the total pressure of the gas, while $f_{\mathrm{i,w}}(p_s, t_{\mathrm{s}})$ is the enhancement factor, which accounts for the non-ideal behaviour of the gas.

The humidity of the gas at the outlet of the generators can also be expressed in terms of frost/dew point temperature, $t_{\mathrm{f,d}}$. The quantity $t_{\mathrm{f,d}}$. represents the temperature at which frost/dew forms when cooling a gas at constant pressure, corresponding to the temperature at which the gas is saturated in equilibrium with ice or water, respectively. In an ideal humidity generator, the saturation temperature, t_s and $t_{\mathrm{f,d}}$. are identical.

The calculation of vapor pressure, $e_{\mathrm{i,w}}$, from the frost/dew point temperature, $t_{\mathrm{f,d}}$, and enhancement factor, $f_{\mathrm{i,w}}(p_s, t_{\mathrm{s}})$, follows the ITS-90 formulations of Hardy [7] in the range -100 °C to 100 °C. For temperatures above 100 °C, vapor pressure calculations are performed in accordance with IAPWS [8], while humidity calculations above 100 °C (saturation vapor pressure via system pressure) follow VDI/VDE 3514 Part 1 [4], which describes the hypothetical saturation state.

The saturation temperature, t_{s}, is measured using platinum resistance thermometers (PRTs), as described in Equation (2):

$$t_s = t_{\mathrm{PRT}}(R_{\mathrm{PRT}}) \quad (2)$$

The saturator pressure, p_{S}, is determined based on the barometric pressure, p_{abs}, and the relative pressure measured at the saturator outlet. In the single-pressure (1P) mode, this outlet pressure is denoted as p_{Sout} (Eq. 3), while in two-pressure (2P) mode, it is referred to as p_{Sat} and is measured after the expansion valve (Eq. 4).

$$\text{In 1P mode}: p_{\mathrm{S}} = p_{\mathrm{abs}} + p_{\mathrm{Sout}} \quad (3)$$

$$\text{In 2P mode}: p_{\mathrm{S}} = p_{\mathrm{abs}} + p_{\mathrm{Sat}} \quad (4)$$

The pressure at the unit under test, p_{UUT}, is determined based on the operating range of the humidity generator. For high-range generator (HRG), where saturation occurs over water, p_{UUT} is given by:

$$p_{\mathrm{UUT}} = p_{\mathrm{abs}} + p_{\mathrm{Sout}} \cdot \frac{p_{\mathrm{UUT}}}{p_s} \quad (5)$$

For low-range generators (LRG), where saturation occurs over ice, the pressure at the unit under test is calculated by Eq. 6, where Δp_{UUT}, represents the pressure measured at the measuring head of the UUT.

$$p_{\mathrm{UUT}} = p_{\mathrm{abs}} + \Delta p_{\mathrm{UUT}} \quad (6)$$

The water vapor partial pressure at the unit under test, e'_{UUT}, is then determined using the following equation:

$$e'_{\mathrm{UUT}} = \frac{e_{\mathrm{i,w}}(t_{\mathrm{s}}) \cdot f_{\mathrm{i,w}}(p_s, t_{\mathrm{s}})}{f_{\mathrm{i,w}}(p_{\mathrm{UUT}}, t_{\mathrm{f,d}})} \cdot \frac{p_{\mathrm{UUT}}}{p_{\mathrm{s}}} \quad (7)$$

Since the vapor pressure equations used in this process cannot be solved analytically for the frost/dew point temperature, $t_{\mathrm{f,d}}$, the value of $t_{\mathrm{f,d}}$is obtained iteratively using Eq. 8.

$$t_{\mathrm{f,d\,S}} = t_{\mathrm{d,f}}(e_{\mathrm{UUT}}) \quad (8)$$

Finally, the relative humidity, $U_{\mathrm{i,w}}$, is calculated as follows:

$$U_{\mathrm{i,w}} = \frac{e_{\mathrm{i,w}}(t_{\mathrm{s}}) \cdot f_{\mathrm{i,w}}(p_{\mathrm{S}}, t_{\mathrm{s}})}{e_{\mathrm{i,w}}(t_{\mathrm{air}}) \cdot f_{\mathrm{i,w}}(p_{\mathrm{UUT}}, t_{\mathrm{air}})} \cdot \frac{p_{\mathrm{UUT}}}{p_{\mathrm{s}}} \cdot 100\ \% \quad (9)$$

4 Acceptance Criteria

The acceptance criteria of the calibration results in primary and secondary calibration methods for frost/dew point temperature and relative humidity of a chilled-mirror hygrometer are presented in Table 1.

Table 1. Acceptance criteria of the calibration results in primary and comparison calibration methods for frost/dew point temperature and relative humidity of a chilled-mirror hygrometer. Note: in this context U_{CMC} refers to the accredited measurement uncertainty.

Acceptance criteria of calibration results
Primary method
· Difference from the monitor chilled-mirror hygrometer: t_f/t_d below -60 °C: ≤ 1/2 U_{CMC} t_f/t_d above -60 °C: ≤ 1/3 U_{CMC} · Standard deviation of primary ref. value: ≤ 20 % U_{CMC} · Standard deviation of the UUT: 25 % U_{CMC} · Repeatability of the UUT: t_f/t_d below -60 °C: ≤ 60 % U_{CMC} t_f/t_d above -60 °C: ≤ 40 % U_{CMC}
Comparison method
· Standard deviation of the reference chilled-mirror hygrometer: ≤ 20 % U_{CMC} · Standard deviation calibration of the UUT: ≤ 25 % U_{CMC} · Repeatability of the UUT: t_f/t_d below -60 °C: ≤ 50 % U_{CMC} t_f/t_d above -60 °C: ≤ 30 % U_{CMC} C

5 Measurement Uncertainty

The main contributions to the measurement uncertainty in primary and secondary calibration methods for frost/dew point temperature and relative humidity of a chilled-mirror hygrometer are presented in Tables 2 and 3, respectively. These uncertainty sources are categorized based on their impact on the reference value and the measured value obtained by the unit under test.

Table 2. Main contributions to the measurement uncertainty in primary calibration methods for frost/dew point temperature and relative humidity of a chilled-mirror hygrometer.

Primary method
Main contributions to the reference value
· Saturation efficiency and stability · Temperature inhomogeneity of the saturator · Determination of the saturation temperature, t_{Sat} · Determination of the saturation pressure, p_{Sat} · Gas pressure at the generator outlet, p_{Sout} · Correlation between pressure and temperature measurement · Contrib. due to the vapour pressure formulation [4,7-8] · For relative humidity calibrations: determination of the chamber/bath temperature and chamber/bath homogeneity and stability
Main contributions to the unit under test indication value
· Repeatability · Resolution · Pressure drop · Flow dependency

Table 3. Main contributions to the measurement uncertainty in comparison calibration method for frost/dew point temperature and relative humidity of a chilled-mirror hygrometer.

Comparison method
Main contributions to the reference value
· Reference value dew point temperature (reference chilled-mirror hygrometer, including; calibration, interpolation between calibration points, repeatability, temperature dependence of the electronics, drift) · Pressure drop · Flow dependency · For relative humidity calibrations: determination of the chamber/bath temperature and chamber/bath homogeneity and stability
Main contributions to the unit under test indication value
· Repeatability · Resolution · Pressure drop · Flow dependency

The calibration and measurement capabilities (CMC) of PI CAL related with chilled-mirror hygrometers are given in Table 4.

Table 4. Calibration and measurement capabilities of PI CAL in related with chilled-mirror hygrometers.

Frost/ dew point temperature	**Expanded Uncertainty (*k*=2)**
Primary method	
[-90, -80[°C [-80, -60[°C [-60, -20[°C [-20, 70[°C [70, 90[°C [90, 95] °C	0.40 K ... 0.20 K(1) 0.20 K... 0.05 K(2) 0.050 K 0.030 K 0.040 K 0.045 K
Comparison method	
[-90, -75[°C [-75, -60[°C [-60, -20[°C [-20, 60[°C [60, 95] °C	0.50 K... 0.20 K(3) 0.20 K... 0.07 K(4) 0.070 K 0.050 K 0.070 K
Relative humidity	**Expanded Uncertainty (*k*=2)**
Primary method	
[1, 98 [%rh	0.10 ... 0.39 K
Comparison method	
[1, 98 [%rh	0.10 ... 0.49 K

Legend: (1) 0.2 K +|t_f+ 80|·0.02; (2) 0.05 K +|t_f+ 60|·0.0075; (3) 0.2 K +|t_f+ 75|·0.02; (4) 0.07 K +|t_f+ 60|·0.0087

6 Conclusion

This work presented a detailed description of the humidity generators used for primary calibration of frost/dew point temperature, along with the key contributors to measurement uncertainty and the acceptance criteria for calibration results. The uncertainty values for the comparison method and relative humidity calibration were also provided.

The Process Insights Calibration Laboratory has the capability to calibrate chilled-mirror hygrometers for frost/dew point temperature using both primary and secondary methods. Primary calibration covers the frost/dew point temperature range between -90 °C and 95 °C, with an expanded measurement uncertainty between 0.4 K and 45 mK, with a coverage factor $k = 2$, while secondary calibration yields to an expanded measurement uncertainty between 0.54 K and 70 mK, with a coverage factor $k = 2$.

Relative humidity calibrations can be performed within the range of 1 %rh and 98 %rh, with an expanded uncertainty between 0.10 %rh and 0.39 %rh for primary calibration and between 0.10 %rh and 0.49 %rh for secondary calibration, with a coverage factor $k = 2$.

These calibration capabilities ensure high accuracy traceability in humidity measurements, supporting industrial and research applications requiring rigorous calibration standards.

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