

The New INTA High-Range Standard Humidity Generator and Its Comparison with the Austrian National Humidity Standard Maintained at BEV/E+E

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Abstract A EUROMET collaborative project has been set up between *Instituto Nacional de Técnica Aeroespacial* (INTA) and E+E ELEKTRONIK Ges.m.b.H, the two designated laboratories of the Spanish and Austrian National Metrology Institutes, *Centro Español de Metrología* (CEM) and *Bundesamt für Eich- und Vermessungswesen* (BEV), respectively. The objective of the project is to provide INTA with a new standard that covers the dew-point temperature range from -27°C to $+90^{\circ}\text{C}$ with a gas flow up to $5\text{L}\cdot\text{min}^{-1}$ in the “two-pressure” mode, extended to 95°C when operated as a continuous flow “single-pressure” generator, and investigate the importance of the enhancement factors in the uncertainty estimations used in support of the participants’ calibration and measurement capabilities (CMC) (The CIPM Mutual Recognition Arrangement, <http://www.bipm.fr/en/cipm-mra/>). The equivalence of the Spanish and Austrian national standards is also to be evaluated, further supporting the outcomes of the Key Comparisons, in which both have already participated. The preliminary results obtained to date are reported and discussed in the context of the project and the consistency of the declared CMC’s.

Keywords Dew-point temperature · Generator · Humidity · Measurement comparison · Standards

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1 Introduction

During the last decade, the Spanish national humidity standards at INTA, using generators based on the well-known two-pressure principle, have been used successfully to maintain and disseminate traceability, and have been compared extensively in the dew-point temperature range from -75°C to $+75^{\circ}\text{C}$. The increased demands on uncertainty and range necessary to satisfy the Institute's aerospace testing requirements, and those of the Spanish accredited calibration laboratories, have made it essential to commission an improved generator. The Austrian national humidity standard at BEV/E+E is based on an improved design of a two-pressure standard humidity generator that represents the current state-of-the-art in this field, and has been successfully intercompared with other leading national standards. It has expanded uncertainties and range, which are consistent with the new requirements of INTA.

The EUROMET collaborative project comprises the following stages: (a) intercomparison of the existing INTA and BEV/E+E generators using a precision optical dew-point hygrometer as a transfer standard over the range from -10°C to 75°C , and the characterization of the transfer standard over the full range at BEV/E+E; (b) the supply to INTA of the main components of a new high-temperature standard humidity generator (HTSHG), of the same basic design as the BEV/E+E generator, and assistance in the assembly and characterization of the unit by INTA; (c) comparison of the two INTA generators using two transfer standards over their common range, followed by measurements using the new generator up to the new extended upper limit; (d) repeated measurements at BEV/E+E over the full range to ensure the long-term stability of the transfer standard during the project and establishment of the revised INTA CMC [1] based on the new generator; and (e) detailed investigation of the influence of enhancement factors on the uncertainty assigned to two-pressure standard humidity generators.

2 Standard Humidity Generators

2.1 BEV/E+E

The BEV/E+E standard humidity generator [2] is based on the two-pressure generator of the Physikalisch-Technische Bundesanstalt (PTB) [3], with a number of design improvements that lead to an extended range and improved stability and flexibility. In this design, the first level of saturation is provided by a downward helix, followed by two condensers stacked horizontally (as opposed to the vertical position of the original PTB design). This has several advantages for condensate level adjustment within the condensers and also for defining a vertical bath gradient to ensure that the final point of saturation is located in the lower condenser, where little heat transfer is required and the immersion conditions are optimum for temperature measurement and control. The measurement of saturator pressure has also been optimized to avoid the well-known problems associated with humid air in contact with precision pressure sensors. The generator can be operated in the two-pressure mode over the range of dew/frost-point temperatures from -80°C to $+90^{\circ}\text{C}$, and from -60°C to 95°C in the single-pressure mode. The measurements of temperature, pressure, and electrical quantities are traceable to the Austrian National Standards.

2.2 INTA

The current INTA standard humidity facilities consist of two-pressure and two-pressure/two-temperature standard humidity generators [4, 5] that combined cover the dew/frost-point temperature range from -75°C to $+75^{\circ}\text{C}$. These generators are used to maintain the Spanish National Humidity Standards and have been used in the CCT-K6 and EUROMET.T-K6 (P621) Key Comparisons in the range from -50°C to $+20^{\circ}\text{C}$.

The new generator components, supplied by E+E, consist of a temperature-stabilized pressure unit, a temperature regulation unit, a pre-saturator, a pressure regulation system, a temperature-stabilized expansion unit, and the main saturator (condenser). An aluminum frame was assembled in order to interconnect the different components and place the saturator in a 95 l temperature-controlled bath (see Fig. 1). This bath is of a larger volume than that used in the E+E generator, and adequately dissipates the heat of the generator for bath temperatures between 0°C to 100°C . The bath was adapted to maintain a constant fluid level to ensure the correct immersion of the transfer lines from the pre-saturator to the first condenser and from the third condenser up to the expansion valve. During the work reported, water was used as the heat transfer fluid in the bath with the INTA generators, and a high-performance perfluoropolyether inert fluid was used by BEV/E+E.

The saturator temperature is measured with two Rosemount 162CE standard platinum resistance thermometers calibrated at INTA at ITS-90 fixed points from the triple point of water to the freezing point of indium, an ASL F700A resistance bridge calibrated in-house using an inductive voltage divider calibrated by PTB, and Tinsley Wilkins standard resistors calibrated by the INTA electricity laboratory. Absolute pressure measurements are performed with Ruska 6230 digital pressure gauges and Paroscientific series 6000 pressure transducers, calibrated by the INTA Pressure and Mass Laboratory using Ruska 2465 pressure balances. All measurements are traceable to recognized National Standards at Centro Español de Metrología (CEM), PTB, and the National Physical Laboratory, UK (NPL). The vapor pressures and enhancement



Fig. 1 Photograph of the new INTA high-range standard humidity generator

factors used in the calculation of the reference dew-point temperature are given in [6–8].

3 Measurements

3.1 Transfer Standards

The transfer standards used in the work reported are precision optical dew-point hygrometers manufactured by MBW ELEKTRONIC AG, of Switzerland, Models DP3-D-BCS-I and DP30-BCS-K2, with serial numbers 92-0322 and 99-1128, respectively. The first unit is the monitoring hygrometer used routinely with the current high-range generator, and the second is to be used as the monitoring hygrometer with the new generator. Both instruments have endoscopes that enable observation of the condensate layer formed on the mirror. The first instrument has a larger diameter mirror and a single-stage Peltier cooler, while the second has a mirror of approximately half the diameter of the first and a two-stage cooler. Another important difference between the units is the condensate layer thickness. The first unit shows optimal control with thicker condensate layers, while the second has a much higher gain control loop and uses a much thinner layer.

Both units have been modified by improving the insulation around the inlet connector on the top of the measurement head and by replacing the exit tube with a stainless-steel temperature-controlled tube, necessary for the optimum performance of the units in the extreme temperature range.

3.2 Pressure Measurements

The measurement of absolute pressure in the saturator and hygrometer is one of the major potential sources of error in high-range humidity measurements using two-pressure standard humidity generators and chilled mirror hygrometers. The difficulty arises from the incompatibility to water vapor of some precision sensors; the errors introduced by unwanted water columns produced in the pressure sampling line, or even in the pressure sensor; and the need to avoid modifying the amount of water vapor in the gas stream applied to the hygrometer. The ideal situation would be to have the pressure sensor at a temperature above the maximum generated dew point (e.g., at 130 °C), but this is incompatible with the level of uncertainty required and the standards available. In the two generator designs contemplated in this work, the approach is slightly different, and this has been taken into account in the comparison.

In the TSC9000, measurement of the saturator pressure is performed at a point close to the final point of saturation at the exit of the last saturator stage via a 4-mm internal diameter stainless-steel tube; the condensate produced during pressurization of the saturator runs back into the saturator. The pressure in the immersed chamber is measured in a similar fashion. The air from the chamber is sampled via a stainless-steel heated hose, with excess flow condensed in a trap and vented to a drain. The pressure at the hygrometer head is defined by the measured chamber pressure, minus the pressure drop in the line produced by the gas flow to the instrument. In the case of the HTSHG and BEV/E+E generators, the saturator pressure is measured via a 10 mm

internal-diameter PTFE line teed into the inlet of the saturator and, thus, the pressure drop in the saturator needs to be evaluated [2].

Validation of the method of pressure measurement was performed by measuring the saturator and chamber pressures with the Ruska sensors at ambient temperature and the heated Paroscientific sensors operating at 40 °C. In order to avoid ingress of moisture into the pressure sensors, the pre-saturator was bypassed during pressurization, ensuring that only dry gas enters the manometers. In this case, the hygrometer reference is obtained by a measurement downstream of the hygrometer in a similar fashion. The reference pressure at the hygrometer head is obtained after correcting for the pressure drop between the hygrometer inlet and outlet, as evaluated over the flow range of interest prior to the commencement of the measurements. Correlation of measurements between the generators was avoided by using independent instrumentation.

3.3 Measurement Method

Measurements were made in the order of increasing dew-point temperatures. In the two-pressure mode, each nominal value of dew-point temperature was generated with at least two different pressure/temperature combinations in order to include the variation due to the enhancement factors [2]. The instrument mirrors were cleaned regularly with isopropyl alcohol, followed by high-purity deionized water, throughout the exercise.

Prior to the commencement of each measurement sequence, at each bath temperature, the presence of sufficient condensate in the two saturator elements was ensured. In the case of the TSC9000, this is obtained through liquid level sensors on the saturator drain and a clear section of tube in the circuit that pumps the excess condensate back to the pre-saturator and, in the case of the new generator, by regularly removing condensate from each stage, at each bath temperature.

In order to establish reproducible and quantified reference conditions, the sample gas flow rate through the hygrometers, as measured after the condensation traps downstream of the instruments, was adjusted to obtain a constant volumetric flow rate at the hygrometer head. This value was set to $0.5 \text{ L} \cdot \text{min}^{-1}$. Failure to observe this procedure can lead to effective flow rates in excess of $2 \text{ L} \cdot \text{min}^{-1}$ at the head at the highest temperatures. All sampling lines were maintained at a temperature 30 °C above the dew-point temperature, with a lower limit of 30 °C.

All measurements of the transfer standard were reported in terms of the independent measurement of the platinum resistance thermometer (PRT) embedded in the mirror assembly of the transfer standard, as prescribed in the existing humidity Key Comparisons.

4 Measurement Results and Discussion

4.1 Comparison of the Two INTA Standards

The measurements performed at INTA on the two transfer standards are depicted in Fig. 2a, b. The results are expressed in terms of the difference between the measured

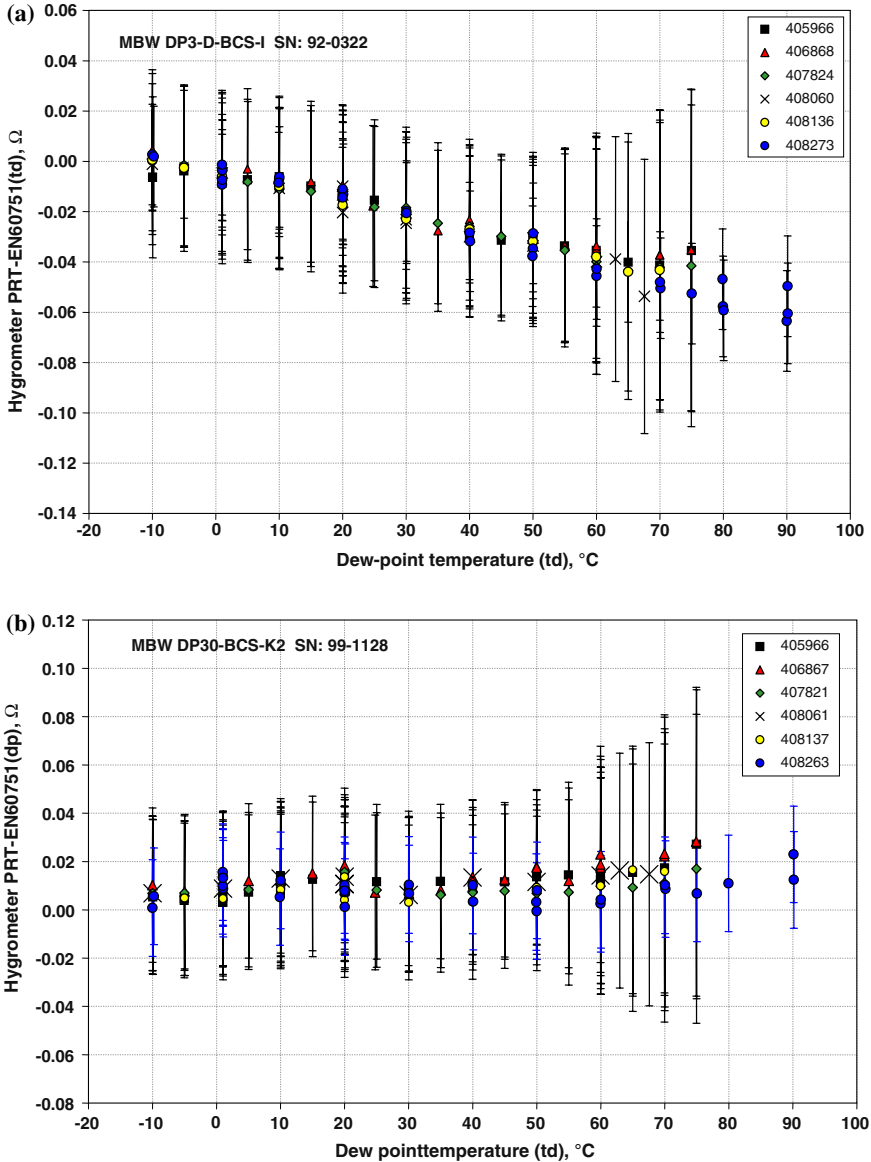


Fig. 2 Deviation of the equivalent temperature, as determined from the mirror PRT resistance, with respect to the applied reference dew-point temperature for transfer standards (a) DP3, S/N: 92-0322 and (b) DP30 S/N: 99-1128, using air. The first four series correspond to the TSC9000 generator, and the last is with the new HTSHG

PRT resistance and the equivalent resistance obtained from the resistance/temperature relationship given in the standard EN 60751 for industrial PRTs, as ordinate, and the reference dew-point temperature as abscissa. One vertical scale division is equivalent to 50 mK. The first four series correspond to the TSC9000 generator and the last to the

Table 1 Differences in the corrections of the two transfer standards at nominal dew-point temperatures in the range from -10°C to $+75^{\circ}\text{C}$, using the existing standard humidity generator (TSC9000) and the new HTSHG

Nominal dew-point temperature ($^{\circ}\text{C}$)	DP30: 99-1128 ^a ($^{\circ}\text{C}$)	DP3D: 92-0322 ^a ($^{\circ}\text{C}$)	Mean ($^{\circ}\text{C}$)
-10	0.016	-0.006	0.005
1	-0.011	-0.002	-0.007
10	0.007	-0.004	0.002
20	0.008	0.002	0.005
30	-0.004	-0.004	-0.004
40	0.009	0.008	0.008
50	0.024	0.006	0.015
60	0.027	0.014	0.020
70	0.022	0.021	0.021
75	0.045	0.038	0.041

^a Difference is reported as Correction (TSC9000) – Correction (HTSHG)

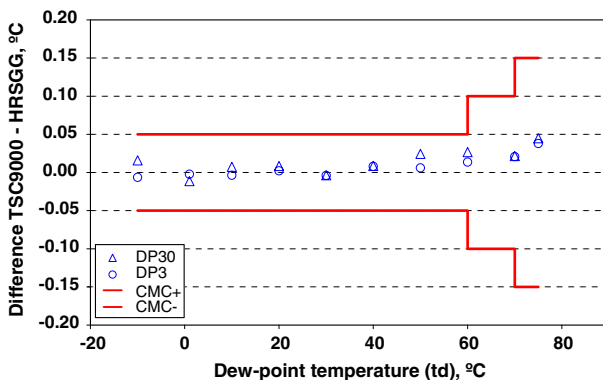


Fig. 3 Differences between the two INTA generators as a function of dew-point temperature in the range from -10°C to $+75^{\circ}\text{C}$, as determined from the transfer standards. The limits are the current CMC entries for INTA

new HTSHG. From these results, the level of reproducibility of the dew-point temperature realization at INTA using the TSC9000 generator and its consistency with the new generator can be established within the limitations of the transfer standards. Table 1 shows the difference between the generators as obtained from the individual transfer standards, and the mean of the two. As can be seen, the level of agreement is excellent from -10°C to $+60^{\circ}\text{C}$, as the values are within the combined reproducibility of the generator and transfer standard, as confirmed by the first four series of results for each hygrometer. At the upper limit of the generator, the difference increases up to twice this amount. All the results are well within the limits of the currently declared CMCs for INTA, as depicted in Fig. 3. The result is particularly satisfactory if we consider that the two generators have saturators of completely different designs and flow rates.

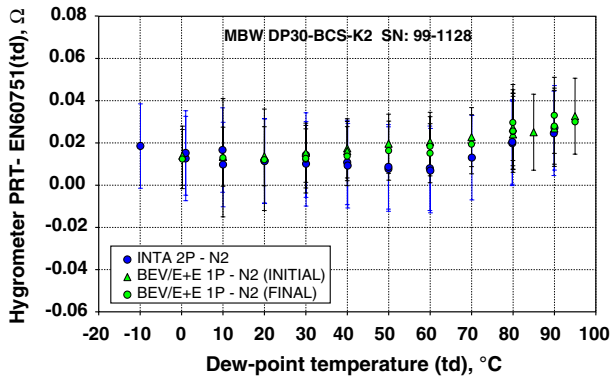


Fig. 4 Deviation of the equivalent temperature, as determined from the mirror PRT resistance, with respect to the applied reference dew-point temperature for transfer standard DP30 S/N: 99-1128, using nitrogen, for the new INTA HTSHG and the BEV/E+E standard—before (INITIAL) and after (FINAL) the INTA measurements

Table 2 Differences in the corrections of transfer standard DP30: 99-1128, using the mean of the two BEV/E+E measurements and the new INTA HTSHG for measurements in nitrogen

Nominal dew-point temperature (°C)	INTA HTSHG (Ω)	BEV/E+E (Ω)	BEV/E+E-INTA (°C)
-10	0.0186	0.0161	-0.006
1	0.0152	0.0132	-0.005
10	0.0132	0.0132	0.000
20	0.0115	0.0129	0.004
30	0.0122	0.0139	0.004
40	0.0100	0.0174	0.019
50	0.0082	0.0180	0.025
60	0.0074	0.0179	0.026
70	0.0130	0.0211	0.020
80	0.0202	0.0264	0.016
90	0.0259	0.0274	0.004

4.2 Comparison of the BEV/E+E and INTA Standards

The measurements at INTA of both transfer standards using the new HTSHG were performed with both nitrogen and air. A small difference, equivalent to approximately 10 mK–15 mK, was detected. This is possibly due to the fact that the air is not free of CO₂, and the enhancement factors are strictly valid for CO₂-free air, but this has not been investigated further.

The results of the measurements performed on the DP30 transfer standard, using nitrogen as the carrier gas, are depicted in Fig. 4. As with the previous figures, the scale corresponds to 50 mK per division on the vertical scale and all the quoted uncertainties and depicted error bars are for expanded uncertainties at a confidence level of approximately 95%. The results confirm the equivalence of the two realizations, with

all the measured values well within the single CMC capabilities of either laboratory. The numerical results are given in Table 2. The maximum difference was observed at a dew-point temperature of 60 °C. The long-term drift of the transfer standards is negligible within the measurement uncertainties, as determined from the difference between the two series measured at E+E and no additional contribution to cover this has been included in the assigned expanded uncertainties.

5 Conclusion

The initial results of a collaborative project between the laboratories holding the Austrian and Spanish National Humidity standards have been presented and discussed. The results show the equivalence of the three generators from -10°C to $+75^{\circ}\text{C}$ and the validity of the new extended range to 90°C with the new E+E-designed high-range standard humidity generator commissioned at INTA, supporting the equivalence of the dew-point temperature realizations of Austria and Spain. Future work on the project will complete the comparison process using both transfer standards and will proceed with the investigation of the influence of the enhancement factors on the uncertainty assigned to two-pressure standard humidity generators, as planned.

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