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A Trial Intercomparison of Humidity Generators at Extremes of Range Using Relative Humidity Transmitters

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Abstract In order to effectively implement the Mutual Recognition Arrangement (MRA) of the International Committee for Weights and Measures (CIPM), national metrology institutes (NMIs) are required to support their claims of calibration and measurement capability (CMC) with a quality system compliant with ISO/IEC 17025, and with suitable evidence of participation in key or supplementary comparisons. The CMC review process, both at regional and inter-regional levels, uses criteria that combine the provisions mentioned above, together with additional evidence demonstrating scientific and technical competence of the institutes. For dew-point temperatures, there are key comparisons in progress under the Consultative Committee for Thermometry (CCT) and under the European regional metrology organisation (EUROMET), together with information available on past regional supplementary comparisons. However, for relative humidity there are, to date, no such comparisons available to support CMC entries. This paper presents and discusses the results of a preliminary investigation of the use of relative humidity and temperature transmitters in order to determine their suitability for the intercomparison of standard humidity generators in support of CMC claims for the calibration of relative humidity instruments. The results of a recent bilateral comparison between 2 NMIs at the extremes of the range up to 98 %rh at 70 °C, and down to 1 %rh at −40 °C are reported. Specific precautions and recommendations on the use of the devices as transfer standards are presented.

Keywords Measurement comparison · Relative humidity · Transmitter

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1 Introduction

Globalization in manufacturing and trade drives an increasing need for international harmonization of measurements. The Mutual Recognition Arrangement (MRA) [1], drawn up by the International Committee for Weights and Measures (CIPM), provides a framework for national metrology institutes (NMIs) to recognize the equivalence of one another's national measurement standards, and of their calibration and measurement certificates. In each field of measurement, equivalence needs to be demonstrated through measurement "key comparisons," providing data on the degree of equivalence between NMIs, which are published online within Appendix B of the CIPM MRA. The comparison results also provide evidence of institutes' calibration and measurement capabilities (CMCs), which are entered into Appendix C of the CIPM MRA.

In the field of humidity measurement and standards, a large proportion of all NMIs have proposed CMC entries for dew-point temperature, and are likely to do so for relative humidity. To evidence these CMC claims, dew-point key comparisons are in progress (one completed [2]). However, comparisons in relative humidity (RH) have only been conducted informally between national laboratories (e.g., [3]), or between laboratories below the national level [4]. As well as the need internationally to substantiate CMC claims, many NMIs also hold accreditations to ISO/IEC 17025:2005 [5], and so need to carry out interlaboratory measurement comparisons to demonstrate proficiency.

In the past, one obstacle to making accurate RH comparisons was the inadequacy of RH instruments for this task, which requires excellent long- and short-term stability, with drift smaller than the uncertainties of the NMI facilities being compared.

In most cases, relative humidity is derived from separate dew-/frost-point and air-temperature measurements. Interlaboratory comparisons in terms of dew point and air temperature can test the realizations of these two quantities. However, realizations of relative humidity in test chambers may potentially suffer errors not easily revealed by conventional comparisons or checks of either dew point or temperature individually. Hence, there is interest in directly testing relative humidity standards, using relative-humidity instruments, through interlaboratory comparisons, if that can be achieved with meaningful accuracy.

For NMI operating standards at particularly high and low relative humidities in wide temperature ranges, the situation is especially uncharted. Few laboratories work at those ranges, and realizations of extreme humidity conditions might seem particularly in need of validation.

In the work reported here, relative humidity transmitters were used on a trial basis to compare standards at the UK National Physical laboratory (NPL) and at the Spanish Instituto Nacional de Técnica Aeroespacial (INTA), operating up to 98 %rh at 70 °C, and down to below 1 %rh at −40 °C. The study had two main aims: to explore how a measurement comparison could be performed at extremes of humidity using relative-humidity sensors; and to test whether two humidity standards could show measurement agreement within the estimated uncertainties at these extremes of range.

2 Standards Facilities Compared

2.1 Overview of Standards and Measurement Setup

In both laboratories, the measurements were conducted in very similar configurations, using dew-point and frost-point generators connected to a test chamber, temperature-controlled inside a climatic chamber. Figure 1 shows a diagram of the NPL test chamber layout, and Fig. 2 shows a photograph of the INTA test chamber. Table 1 summarizes the normal operational ranges and uncertainties of the NPL and INTA relative-humidity facilities.

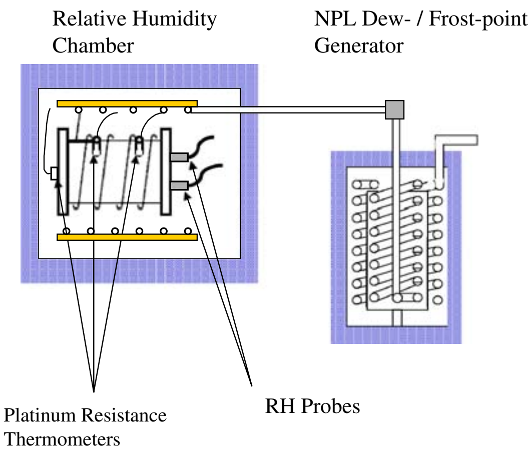

Fig. 1 Schematic diagram of the NPL primary relative humidity standard and measurement configuration

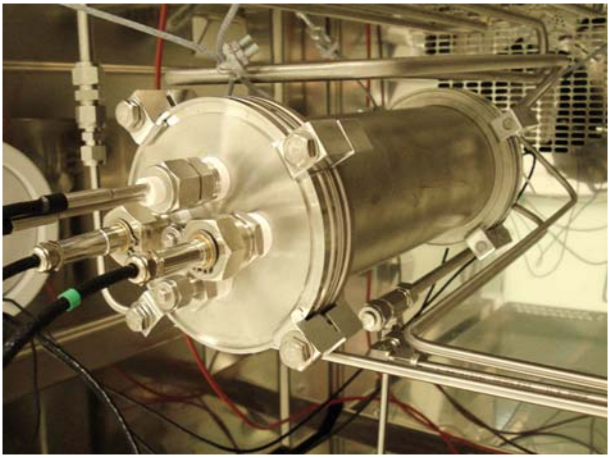

Fig. 2 Photograph of the INTA relative humidity test chamber

Table 1 Normal operating range and uncertainty of relative humidity standards of INTA and NPL

Laboratory	relative humidity range	Best measurement capability ($k = 2$)
INTA	1 %rh to 98 %rh at air temperatures from −40 °C to +70 °C	0.2 %rh to 1.0 %rh
NPL	2 %rh to 98 %rh at air temperatures from −40 °C to +100 °C	0.6 % of reading + 0.1 %rh

2.2 Details of NPL Relative Humidity Standard

For this work, the NPL relative humidity realization was housed inside a Montford Model HC-500-R climatic chamber of volume 0.125 m^3, used for temperature control only. The relative humidity (inner) test chamber is an internally electropolished stainless steel cylinder of diameter 65 mm and length 300 mm (capacity nominally 1 L). This is shielded by a surrounding thick brass cylinder with a nominal diameter of 180 mm, to increase temperature stability and uniformity. Sample gas from a primary single-pressure dew-point or frost-point generator [6] flows at $1\,L \cdot min^{-1}$ through 2 m of internally electropolished stainless steel tubing coiled inside the brass shield to condition the humidified gas to the selected temperature before entering the inner test chamber. Air temperature is measured using five PRTs distributed within the inner test chamber. A pressure sensor connected to the chamber is used to correct the generated frost/dew point for any pressure difference between the primary humidity generator and the chamber (of greatest significance at highest dew points). A calibrated condensation hygrometer is used to monitor the gas exiting the chamber, as a consistency check.

2.3 Details of INTA Relative Humidity Standard

For the work carried out at INTA, measurements for temperatures above 4 °C were performed in the immersed chamber of the INTA high-range standard humidity generator [7]. The remaining measurements were performed in a calibration system comprising humidity generators purely as a stable humidity source, together with an external measurement chamber enclosed in a Vötsch VT 7034 climatic chamber to provide the ambient-temperature conditions. The reference relative humidity measurements are obtained with MBW optical dew-point hygrometers, calibrated using the national standard humidity generators [7] and platinum resistance thermometers (PRTs). The inner chamber is constructed using standard vacuum components and electro-polished stainless steel tubes and fittings. The chamber has an internal diameter of 100 mm and is 260 mm in length. The output of the humidity generator is fed to the inner chamber via a heat exchanger and is introduced through the back flange of the chamber. The air also exits the chamber through this flange, via a tube that protrudes to within 10 mm of the front flange, ensuring complete air changes. Also, via the back flange, separate samples of the gas are taken from the center of the chamber to an absolute pressure transducer and the hygrometer with suitable flow rates to equalize the pressure drops. Two PRTs are inserted, together with the relative humidity probes, through the front flange, and a third sensor is introduced via the back flange to different immersion depths in order to obtain an indication of both the radial and axial temperature homogeneity. The flow rate through the chamber is optimized at the lowest frost-point temperature, and was set to $1.7\,\mathrm{L} \cdot \mathrm{min}^{-1}$ throughout the reported measurements.

3 Measurements

3.1 Overview of the Measurements

The comparison was carried out over a period from 2002 to 2007 in three stages: high range (+70 °C), mid-range (+23 °C), and low range (0 °C and −40 °C). Different instruments were used for each stage. A pair of hygrometers was used simultaneously for the extreme range measurements and a single instrument for the measurements at 23 °C. The use of two nominally identical transfer standards provides not only a degree of redundancy (essential in case of anomalous behavior of one of the devices), but also offers additional information through observing the performance of the sensors relative to each other [8].

As this was only a trial, with just two participants, a measurement protocol was not formalized, but both laboratories followed their local routine procedures based on considerable experience and accepted good practice. Suitable measures were taken to ensure confidentiality of the results while measurements were in progress.

At 23 °C and above, straightforward comparison measurements were made by comparing the hygrometers against the reference in each laboratory. More extensive measurements were made in the low-temperature and low-humidity range, because this was considered more challenging and less well studied.

3.2 Transfer Standards

The instruments used for the comparison were polymer-based relative humidity transmitters (i.e., humidity sensors with signal processing giving an output in DC voltage). The details of the instruments used are given in Table 2. The resolution of the voltage outputs of the instruments was equivalent to nominally 0.01 %rh.

Before any results were obtained, all instruments were cycled several times over the relevant sub-range of the comparison. The performance of the low-range instruments (Serial Nos. T5050025 and T5050027) was characterized in some detail during the

Table 2 Details of the relative humidity transfer standards used

Temperature	Make	Model	Serial number
−40 °C to 0 °C	VAISALA	HMP238	T5050025
−40 °C to 0 °C	VAISALA	HMP238	T5050027
23 °C	ROTRONIC	Hygromer	16705 001
70 °C	VAISALA	HMP238	T5050021
70 °C	VAISALA	HMP238	T5050022

course of the comparison measurements. This included evaluation by INTA of the temperature dependence of the relative characteristic, and some evaluation of long-term stability of the instruments, through repeated measurements by NPL.

3.3 Measurement Method for the Comparison

Comparison measurements were made in rising order of humidity values at each set temperature, and then the cycle repeated. The probes were always used fully immersed in gas at the test temperature, to avoid stem conduction errors [9]. At high humidities, trace heating was used on gas sample lines from the dew-point generators as needed to avoid condensation. At low humidities, due precautions were taken against leaks and desorption, using suitable connectors and purge times. For the low temperature measurements performed at NPL, the sintered filters covering the sensors were removed to minimize response times and avoid any influence of contamination of the filter assemblies.

Hygrometer settling times varied considerably at different parts of the range with the greatest at the low extreme. A time of not less than 2 h was allowed for stabilization of the instrument and up to 24 h at the lowest range until a set criterion for stability had been satisfied. At this point, a set of at least 10 voltage readings from each instrument was taken over a period of between 20 min and 1 h, along with all the measurements from the standards.

For saturation temperatures below 0 °C, relative humidity values are reported with respect to supercooled water, as typically indicated by relative humidity sensors.

4 Results

4.1 Instrument Characterization

Temperature characterization of the humidity performance of the two instruments used in the low range (T5050025 and T5050027) is shown in Fig. 3. A humidity of 10 %rh was applied during 10 °C steps in air temperature from −40 °C to +20 °C, and the sensor readings were compared against a reference relative humidity value derived from dew-point and air-temperature measurements. The sensor calibration correction was found to vary linearly by approximately 0.10 %rh · $°C^{-1}$ below 0 °C. Above 0 °C, the correction varied linearly by approximately 0.03 %rh · $°C^{-1}$. This was practically identical for both hygrometers. This determines (for these hygrometers) the uncertainty incurred in deviating from the nominal temperatures agreed for comparison measurements.

The stability of the instruments T5050025 and T5050027 in the low-temperature range was monitored over the period of the comparison. The difference between the hygrometers and the NPL standard showed some long-term variability, approaching ±0.5 %rh at 15 %rh. The difference between the pair of instruments also showed some progressive drift. At 15 %rh and −40 °C, the between-pair difference gradually increased from approximately 0.03 %rh during the first NPL measurements to 0.11 %rh during the next NPL measurement, then to 0.27 %rh at INTA, and to 0.42 %rh during

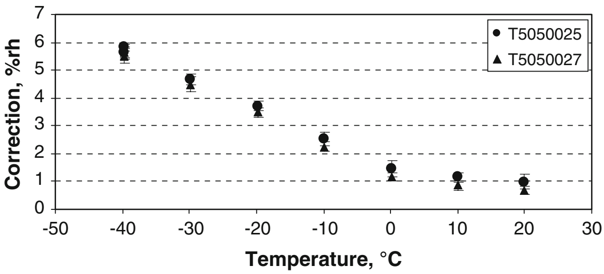

Fig. 3 Graph showing variation in humidity calibration correction at 10 %rh, in the temperature range from −40 °C to +20 °C, for hygrometers T5050025 and T5050027. Error bars represent the expanded uncertainties of the measurements at a coverage factor of $k = 2$

the final NPL measurements. The absolute values of the results suggested one sensor had drifted more than the other. From these observations, the worst-case contribution of hygrometer irreproducibility to the uncertainty of the comparisons at −40 °C was estimated to range from ±0.15 %rh (near 1 %rh) to ±0.5 %rh (near 15 %rh). At 0 °C, the reproducibility of the instruments appeared similar, so the same uncertainties were applied. This was incorporated as an uncertainty in the comparison between the INTA and NPL standards.

At the lowest humidity values measured, the instruments were observed to "bottom out" at a limit of operation of approximately 0.4 %rh.

4.2 Interlaboratory Comparison Measurement Results

Figure 4a, b, and c shows graphs of the difference (reference minus hygrometer reading). Each result plotted was the mean of at least 10 readings of two hygrometers (except at 23 °C where only one hygrometer was used). On Fig. 4a and b, the NPL values are results obtained at different times, with some scatter illustrating the combined reproducibility of the standard and hygrometers. The error bars show the estimated uncertainties of the measurements at a coverage factor $k = 2$ (approximately 95 % confidence level) combining in quadrature the individual assigned expanded uncertainties. The error bars include the uncertainty due to the performance of the hygrometers at the time of measurement, but not their longer-term stability.

In this context, the absolute values of difference from the reference are not of particular significance—the matter of interest is how closely the NPL and INTA estimates agree. To allow comparisons to be made between results at slightly different humidity values, curves were fitted to the results of each lab at each temperature and these are also shown in Fig. 4a and b. The data below 15 %rh were treated together for curve fitting—i.e., the curves in Fig. 4b are continuous with those in Fig. 4a.

For 0 °C and below, using the best-fit curves, estimated (interpolated) values were derived for exact nominal humidity values of 1 %rh, 5 %rh, 10 %rh, and 15 %rh. For the measurements at 23 °C and above, the results were compared directly (without curve fitting and interpolation). From this information, inter-laboratory differences were calculated, and these are summarized in Table 3. The table shows the difference

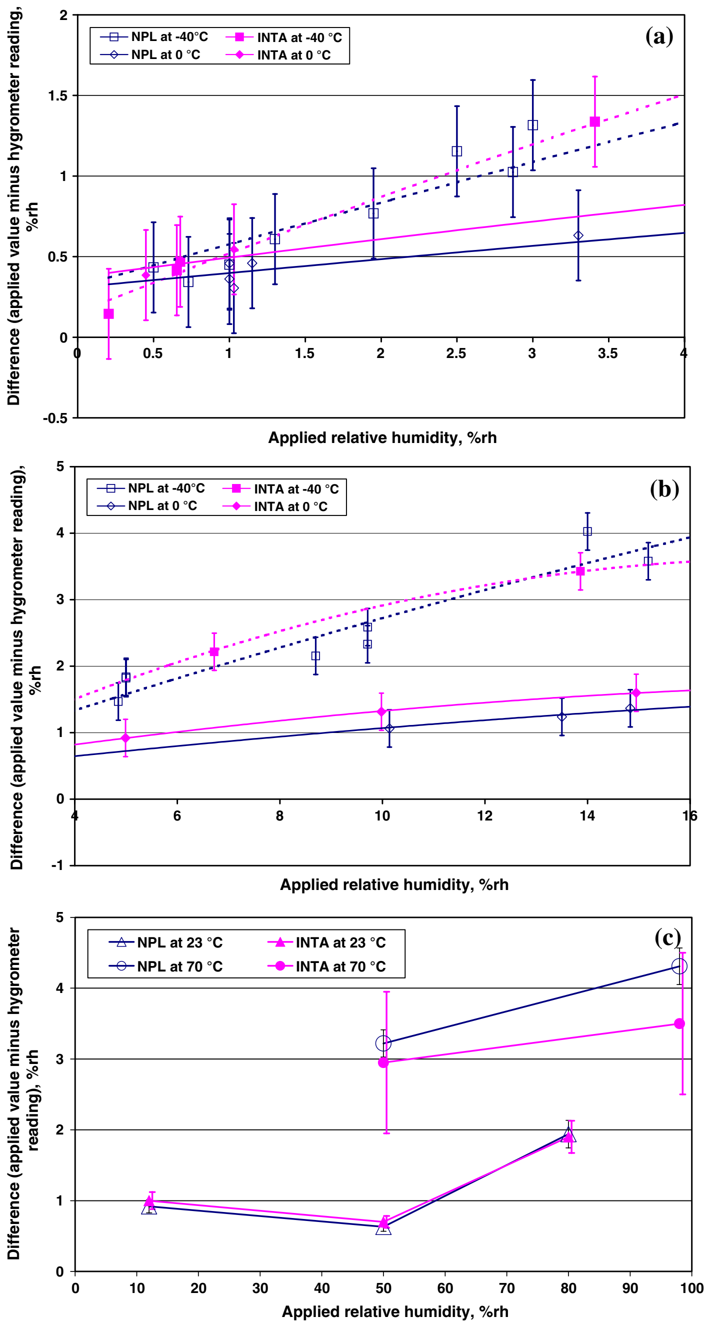

Fig. 4 Results of interlaboratory comparison measurements, expressed as applied reference value minus hygrometer value: (**a**) humidities below 4 %rh, at 0 °C and −40 °C, (**b**) humidities from 5 %rh to 15 %rh, at 0 °C and −40 °C, and (**c**) a wide humidity range, at 23 °C and 70 °C. Uncertainties of the measurements at a coverage factor $k = 2$ are shown by error bars (some slightly offset for clarity). Curves are fitted to results in (**a**) and (**b**) to give best estimates from scattered data

Table 3 Results of relative humidity intercomparison measurements at a range of temperatures and humidities, expressed as difference between laboratories (INTA minus NPL) in percent relative humidity

Relative humidity (%rh)	−40 °C		0 °C		+23 °C		+70 °C	
	INTA-NPL (%rh)	U (%rh)	INTA-NPL (%rh)	U (%rh)	INTA-NPL (%rh)	U (%rh)	INTA-NPL (%rh)	U (%rh)
1	−0.02	±0.19	+0.10	±0.19	–	–	–	–
5	+0.21	±0.28	+0.19	±0.28	–	–	–	–
10	+0.16	±0.57	+0.25	±0.57	–	–	–	–
15	−0.21	±0.60	+0.25	±0.60	+0.08	±0.72	–	–
50	–	–	–	–	+0.07	±0.98	−0.15	±1.08
80	–	–	–	–	−0.04	±1.16	–	–
98	–	–	–	–	–	–	−0.40	±1.13

Differences are shown for nominal values of 1 %rh, 5 %rh, 10 %rh, 15 %rh, 50 %rh, 80 %rh, and 98 %rh, based on interpolations of best-fit curves to results at −40 °C and 0 °C, and direct subtraction at higher temperatures. The expanded uncertainties, U, at a coverage factor $k = 2$, are the combination of the laboratory uncertainties for the measurements (together with allowances at −40 °C and 0 °C for hygrometer long-term stability)

(INTA-NPL) and the uncertainty of this difference, including an uncertainty allowance for instrument long-term stability as described in Sect. 4.1 above.

5 Discussion

At 23 °C, from 10 %rh to 80 %rh, the measurements by INTA and NPL agreed to within the quoted accredited uncertainties for either laboratory individually, which is a completely satisfactory outcome. At 70 °C, at 50 %rh and 98 %rh, the results showed agreement within the combined $k = 2$ uncertainties estimated by INTA and NPL. At both 23 °C and 70 °C, the reported uncertainties do not include allowance for any long-term instability of the sensors. In view of this, the agreement within the uncertainties is a good outcome.

Many more measurements were made for the low-temperature and low-humidity part of the comparison, because this was less explored, and was expected to be more difficult. In fact, good agreement was also observed in this range. The best-fit curves for each laboratory do not differ from each other at any point by more than the quoted $k = 2$ uncertainty of either laboratory. At the lowest relative humidities, below 5 %rh, agreement between NPL and INTA results was well within the uncertainties shown in Fig. 4a. This suggests firstly that the hygrometers easily performed reproducibly enough for this type of comparison in this range. Secondly, this suggests that the uncertainties quoted by the laboratories below 5 %rh could be construed as conservative, since they easily covered both the scatter and systematic differences observed in the comparison. In principle, the combined uncertainty of multiple results (where the simultaneous average from a pair of sensors was used) should take into account correlation in the uncertainties; not doing so can be expected to lead to conservative (overestimated) uncertainty values.

Overall, there appear to be regions where the difference between NPL and INTA is systematic (e.g., INTA higher than NPL at 0 °C and lower than NPL at 70 °C).

However, this is not the case at the other temperatures, and so it would be difficult to interpret this as a significant trend.

The typical slope of the fitted curves shows that the calibration correction of the sensors at −40 °C changes by up to 0.3 %rh per 1 %rh of applied humidity. This is potentially more significant than the temperature sensitivity (0.1 %rh · $°C^{-1}$), but in this case is largely taken into account by the curve fitting and interpolation.

It is relevant to note that at −40 °C and 5 %rh, an uncertainty of around ±0.3 %rh (as reported in Table 3) corresponds to roughly ±0.4 °C in either air temperature or frost point (at a frost point of about −64 °C). At −40 °C and 1 %rh, an uncertainty of ±0.2 %rh (as in Table 3) corresponds to about ±1 °C (at a frost point near −75 °C). This would be a consideration in deciding whether an interlaboratory comparison in dew point or in relative humidity would provide the more stringent and feasible test of measurement capability.

For the measurements at 98 %rh, with hindsight it would have been advisable to fully test that the hygrometers had not reached a "ceiling" at their limit of measuring range, by checking the instrument sensitivity around this value. The corresponding check was made in the low-range measurements, where the lower operating limit was established at 0.4 %rh, as reported.

The instruments selected for this work were models available at the time of purchasing. However, by the end of the study at least one new model for use at very low humidities became available, and might be a better choice if similar work were repeated in the future.

In this work, there was no agreement whether to measure with the sensor filters on (INTA measurements) or off (NPL measurements). This clearly affected sensor response time, but it could potentially also have affected readings if contamination was present on the filters (filter contamination is likely to lead to sensor under-reading). Although there was no particular evidence that this affected the work reported here, this would be a useful area for future study, and this aspect of the procedure should be specified in any formal comparison using these types of sensors.

Measurement of air temperature, although an essential part of relative humidity realization, was not explicitly a part of this study.

6 Conclusion

The comparison using relative humidity transmitters showed very good agreement between INTA and NPL relative humidity standards at low relative humidities at −40 °C and 0 °C. Good agreement was also observed at 23 °C and at 70 °C through a wider range of relative humidities.

The outcome of this trial intercomparison provides evidence in support of the measurement capabilities of INTA and NPL within their relevant scopes of accreditation to ISO/IEC 17025:2005 [5]. This work demonstrates the potential suitability of relative humidity sensors for a comparison of this kind. In particular, the feasibility of this at low temperatures and relative humidities has been shown (subject to the performance of the particular hygrometers used).

This work has highlighted some precautions to be taken when using relative hygrometers for interlaboratory comparisons, particularly at extremes of range. In such comparisons, it is advisable to test the limits of operation as part of the prior characterization of the instruments (and perhaps again after the comparison) to be completely sure that valid measurements at extreme values can be made. In interlaboratory comparisons, temperature and humidity sensitivity coefficients of hygrometers should be evaluated in advance; this can determine how closely participants must adhere to the nominal values for comparison.

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