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An Interlaboratory Comparison of Relative Humidity and Temperature Probes Using Diverse Generation Techniques

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Abstract An interlaboratory comparison using relative-humidity (RH) and temperature probes at three national measurement institutes and two accredited laboratories has been carried out. The work had three purposes: firstly, to establish the instruments' level of reproducibility and suitability for use as transfer standards within their specified range of operation; secondly, to show the agreement of a method of RH generation utilizing certified non-saturated salt RH standards when compared with a method of RH calibration using a chilled-mirror reference and platinum-resistance thermometers; and finally, from the results obtained it is possible to establish the equivalence between the participating laboratories, to the level of uncertainty achievable with the transfer standards used. A total of six RH probes were tested in two groups. The instruments of the first group were calibrated in the range from 10 %rh to 90 %rh at a temperature of 23 °C. The second group of instruments was measured in the same RH range, but at the temperatures of 5 °C, 23 °C, and 50 °C. The objective of the tests on the second group of instruments was to determine the effect of a wider operating temperature range on performance. This article presents and discusses the results of the

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comparison in the context of an international collaboration that provides confidence in the measurements performed by the participants within their respective accredited scopes and the ILAC or the CIPM mutual recognition arrangements.

Keywords Comparison · Interlaboratory · Relative humidity

1 Introduction

Interlaboratory comparisons are proficiency testing activities that provide a quantifiable means of providing confidence in the quality of calibrations performed. While in many measurement areas there is extensive experience in the organization of interlaboratory comparisons; in the field of humidity, there is limited international experience involving accredited laboratories [1].

One of the main difficulties in performing such exercises in the past can be attributed to the inherent limitations of the available transfer standards and the increasingly improved measurement capabilities of the calibration laboratories. In order to establish the fitness for purpose of current commercially available instruments, the Swiss manufacturer, Rotronic AG, decided to organize an interlaboratory comparison between several leading European laboratories with recognized experience in the calibration of relative-humidity (RH) sensors.

All participants form part of the internationally recognized metrology and calibration infrastructure, via the ILAC [2,3] or CIPM [4] mutual recognition arrangements that ensure traceability to recognized national standards and the implementation of quality systems to ISO/IEC 17025:2005.

2 Measurements

2.1 Transfer Standards

For this exercise, six RH and temperature probes were used as transfer standards (see Table 1). Each probe was connected to a Rotronic HygroLab 2 display instrument. The output from each probe was monitored and logged via an RS-232 signal to a PC running the Rotronic HW3 software. The instruments were split into two groups; the first group comprised two sensors of different models, a HygroClip S (for general

Table 1 Transfer standards and measurements performed

Group	Model	Serial number	Measurements
1	Hygroclip S	37251 166	10 %rh–90 %rh at 23 °C
	Hygroclip S3	35197 035	
2	Hygroclip S	37251 187	10 %rh–90 %rh at 5 °C, 23 °C, 50 °C
	Hygroclip S3	35197 036	
	Hygroclip IC1	36702 002	
	Hygroclip IC1	36702 005	

use) and a HygroClip S3 (for meteorology applications), and was to be calibrated at laboratory ambient temperature only. The second group of instruments comprised one each of the HygroClip S and HygroClip S3 as in group 1 and two of the model HygroClip IC-1 (especially well suited for high-temperature applications) and was to be calibrated across an extended temperature range.

2.2 Measurement Protocol

Prior to commencent of the exercise, the instruments were factory adjusted according to Rotronic AG Switzerland procedures and no further adjustments were performed throughout the exercise. The suitability of the devices was then determined by their characterization [5] at the Rotronic AG Switzerland SCS accredited laboratory and the INTA temperature and humidity laboratory which performed measurements at 23 °C to give an indication of repeatability, the results of which are discussed in Sect. 3.2. The instruments were cycled through the calibration points 50 %rh, 10 %rh, 50 %rh, 90 %rh, and 50 %rh in five cycles.

The agreed measurement protocol established fixed nominal measurement points and the order of measurement (to reduce the effects of hysteresis) and required participants to follow their own in-house procedures based on extensive experience and good measurement practice [4,6,7].

The instruments in group 1 were to be calibrated at each laboratory in turn at eleven RH calibration points at 23 °C (50 %rh, 10 %rh, 35 %rh, 50 %rh, 75 %rh, 90 %rh, 75 %rh, 50 %rh, 35 %rh, 10 %rh, and 50 %rh), in ascending and descending RH to account for the hysteresis of the probes. The four instruments in group 2 were to be subjected to the same series of RH points at the temperatures of 23 °C, 5 °C, 50 °C, and again at 23 °C.

The drift during the comparison was to be estimated from repeated measurements at INTA. Intermediate tests were also performed by Rotronic AG.

2.3 Traceability

The methods of calibration and the traceability to internationally recognized national standards are summarized in Table 2. At Rotronic AG, measurements were performed

Table 2 Participants, measurement schedule, method of calibration, and traceability

Laboratory	Dates of measurement	Method	Traceability
Rotronic AG (CH)	March–June 2005	Comparison	METAS
INTA (ES):	June–November 2005	Comparison	INTA
NPL (GB):	Janaury–February 2006	Comparison	NPL
PTB (DE):	March 2006	Comparison	METAS
Rotronic AG (CH)	October 2006	Comparison	METAS
INTA (ES)	November 2006	Comparison	INTA
Rotronic UK Ltd. (GB)	January 2007	Comparison	NPL
Rotronic UK Ltd. (GB)	February 2007	Unsaturated salt solutions	METAS via Rotronic AG

using a two-pressure humidity generator and a chilled-mirror hygrometer. At INTA and NPL, they were performed within their respective scopes of accreditation [1] by comparison with the air-temperature and dew-point working standards. The measurements at PTB were performed directly with the standard humidity generators that are the basis for the German national standards calibration measurement capability (CMC). At Rotronic UK, measurements were performed using two different methods: (a) using non-saturated salt solutions [8], in a temperature-controlled laboratory (23 °C ± 2 °C), covered by the scope of the laboratory's existing UKAS accreditation (instruments in group 1 and initial measurements at 23 °C for group 2), and (b) in a two-pressure generator by comparison with a chilled-mirror hygrometer and calibrated PRTs (remainder of points in group 2), outside the scope of accreditation.

3 Measurement Results and Discussion

The diversity of probes and the differing severity of the climatic conditions to which the instruments were subjected in groups 1 and 2 enable a number of considerations to be addressed in the context of the suitability of the probes for intercomparisons as well as the degree of equivalence among the participating laboratories.

3.1 Repeatability and Reproducibility

The repeatability of all of the instruments used as part of the interlaboratory comparison was evaluated as part of the initial characterization carried out by Rotronic AG Switzerland and INTA and was found to be within ±0.2 %rh at 23 °C in the range from 10 %rh to 90 %rh. The reproducibility of the instruments in both groups was evaluated for all three of the model types used as part of the comparison. The difference between readings of instruments of the same model type was calculated at each of the 50 %rh points during the calibration runs completed at 23 °C (including data from Rotronic UK completed using non-saturated salt solutions), 5 °C, 50 °C, and the final 23 °C run. The reproducibility (including hysteresis—see Sect. 3.3) for each of the sensors is presented in Figs. 1–4. The reproducibility has been defined as the modulus of the semi-interval that encompasses all the measurements at 50 %rh with respect to the mean value, for each laboratory, in order to establish an upper bound for this contribution.

Figure 1 shows the results for sensors of the same models (S and S3) from groups 1 and 2 that have undergone different temperature and humidity cycling in order to determine if, for a given model instrument, performance is improved by limiting the measurements to 23 °C. The results clearly demonstrate that, at 23 °C, the reproducibility of the probes of group 1 is superior to those of group 2 (for the same models) that were subjected to harsher conditions. This supports the idea that, in a comparison, it is worth limiting the measurement range to match the measurement capability being tested.

Figures 2–4 show the results for the four instruments of three different models in group 2 that underwent the full thermal/humidity cycling, for 23 °C, 5 °C, and 50 °C, respectively. All the instruments exhibit similar results with no apparent advantage of

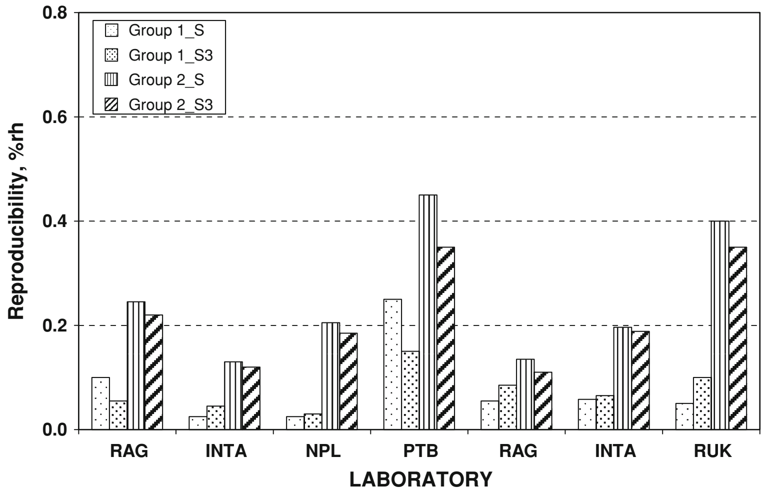

Fig. 1 Reproducibility for HygroClip-S and S3 instruments of both groups at 23 °C, obtained from all the measurements reported at 50 %rh (includes hysteresis)

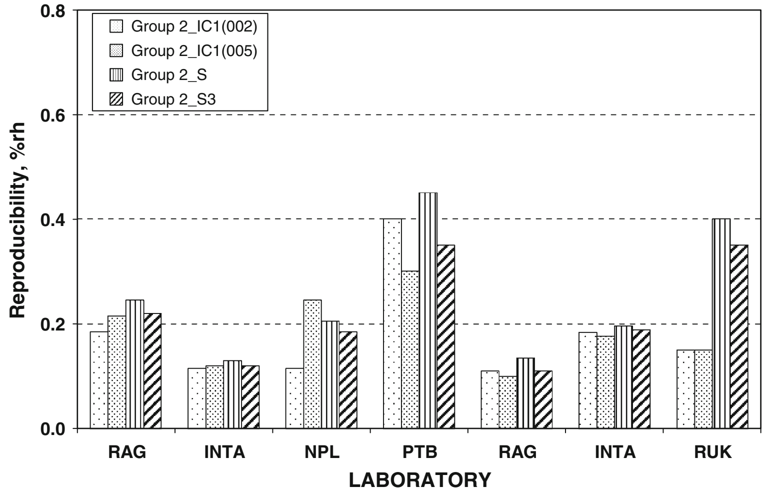

Fig. 2 Reproducibility for HygroClip-S, S3, and IC-1 instruments of group 2 at 23 °C, obtained from all the measurements reported at 50 %rh (includes hysteresis)

the more rugged model IC-1 with respect to models S and S3 under the same conditions in the range reported.

3.2 Long-term Stability

The long-term stability (drift) of all instruments during the 2-year period of the comparison, as evaluated from the repeat measurements at INTA, is reported in Figs. 5–8.

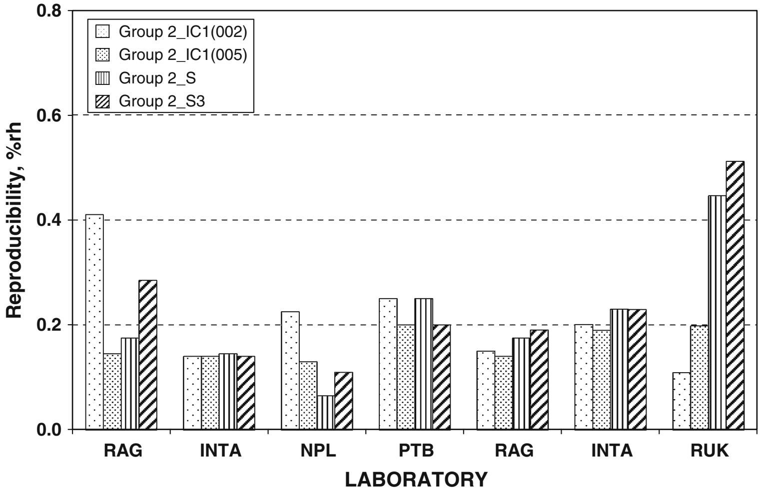

Fig. 3 Reproducibility for HygroClip-S, S3, and IC-1 instruments of group 2 at 5 °C, obtained from all the measurements reported at 50 %rh (includes hysteresis)

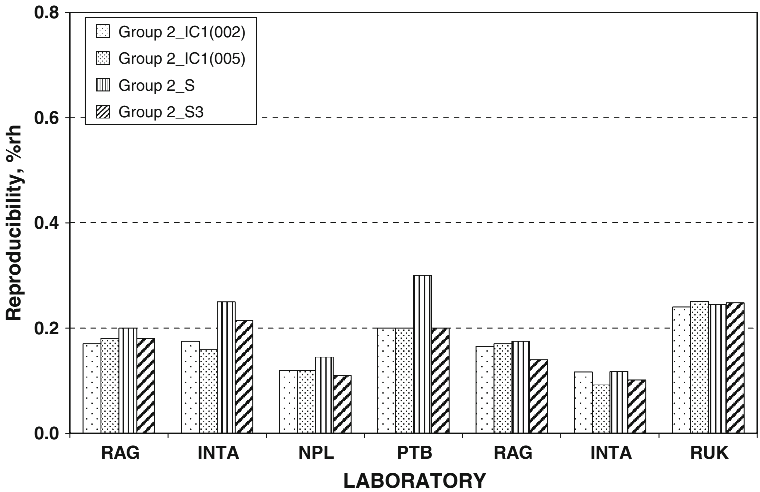

Fig. 4 Reproducibility for HygroClip-S, S3, and IC-1 instruments of group 2 at 50 °C, obtained from all the measurements reported at 50 %rh (includes hysteresis)

The results are the mean of the reported differences at each nominal relative humidity. At 50 °C, the industrial-type probes (IC-1) show a better stability, as expected. The results at 5 °C show the opposite from what was expected—the industrial type instruments are specifically designed for use in the higher temperature ranges. In any case, all the results are consistent with the instrument specifications and the expanded uncertainty of measurement.

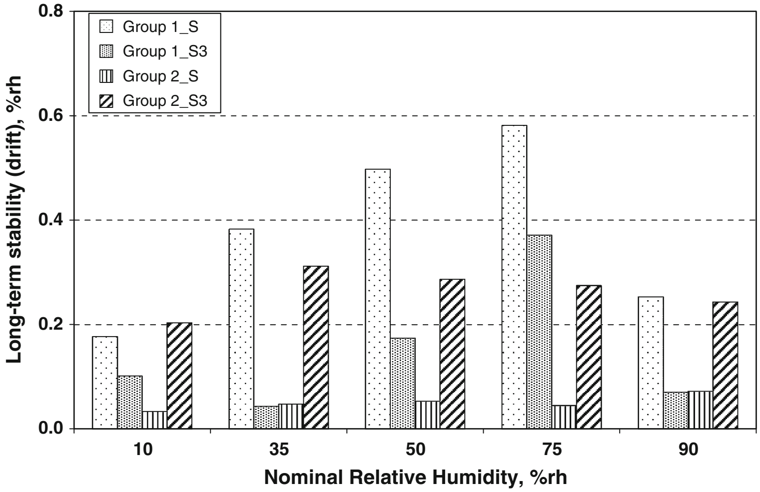

Fig. 5 Long-term stability for HygroClip-S and S3 instruments of both groups at 23 °C

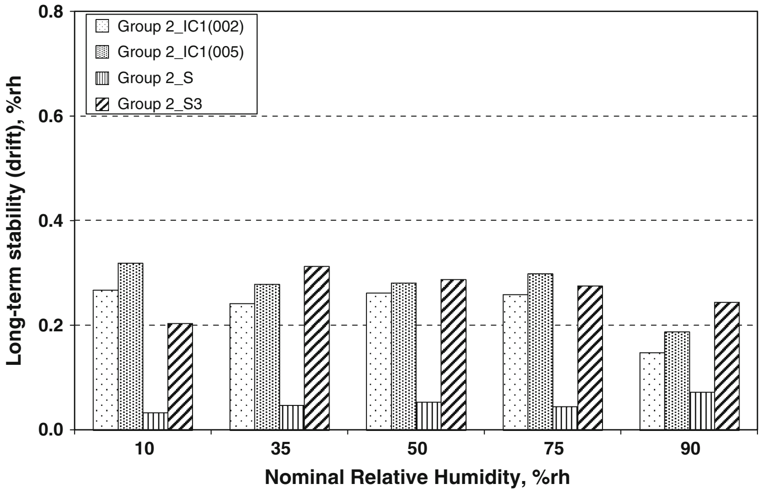

Fig. 6 Long-term stability for HygroClip-S, S3, and IC-1 instruments of group 2 at 23 °C

3.3 Hysteresis

The hysteresis of the instruments was evaluated as the difference between the deviation from the reference at the 50 %rh calibration point on the "upward" run and the deviation from the reference at the 50 %rh calibration point on the "downward" run at all temperatures for all six instruments. In all cases (for both groups of instruments), the hysteresis was seen to be <0.9 %rh (a semi-interval of ±0.45 %rh). Capacitive-type RH instruments are susceptible to hysteresis, and this value is deemed to be most satisfactory. It is because some hysteresis was expected that the comparison was designed

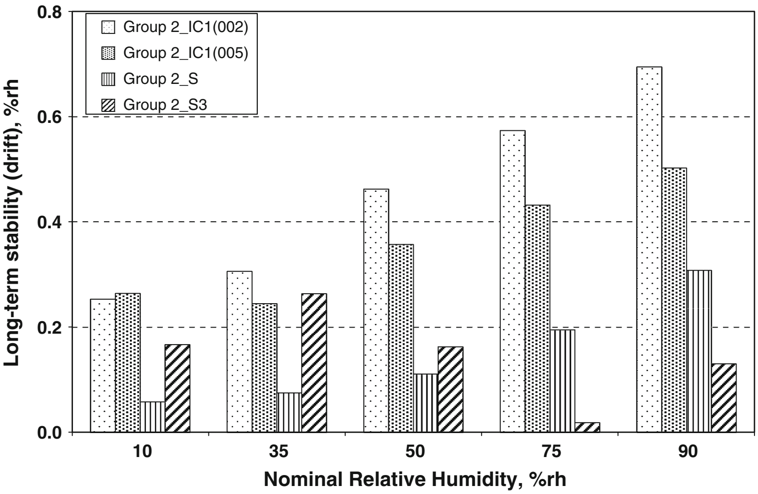

Fig. 7 Long-term stability for HygroClip-S, S3, and IC-1 instruments of group 2 at 5 °C

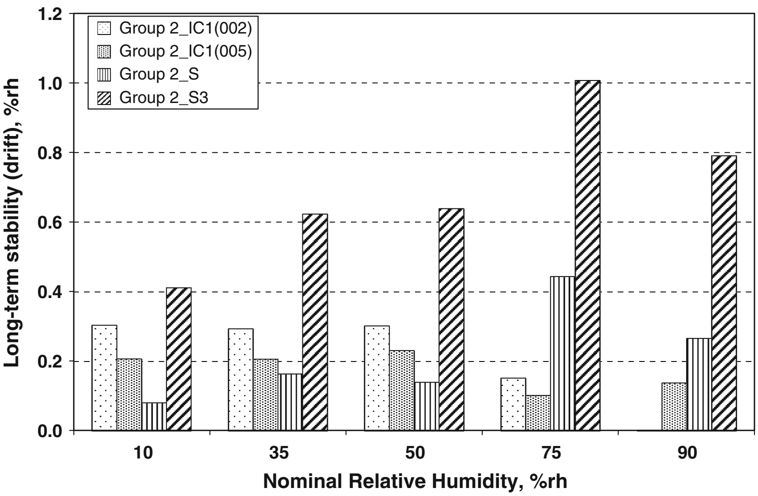

Fig. 8 Long-term stability for HygroClip-S, S3 and IC-1 instruments of group 2 at 50 °C

so that all laboratories performed all of the measurements in the same order, allowing the degree of equivalence to be determined from measurements in the same direction.

3.4 Degree of Equivalence

The main characteristics of all six probes have been reported in the previous sections in terms of their repeatability, reproducibility (including hysteresis), and long-term stability during the period of the comparison. The use of multiple transfer standards has the benefit that the optimum transfer standard can be used to evaluate the degrees of equivalence in each measurement range. In the results reported, however, three

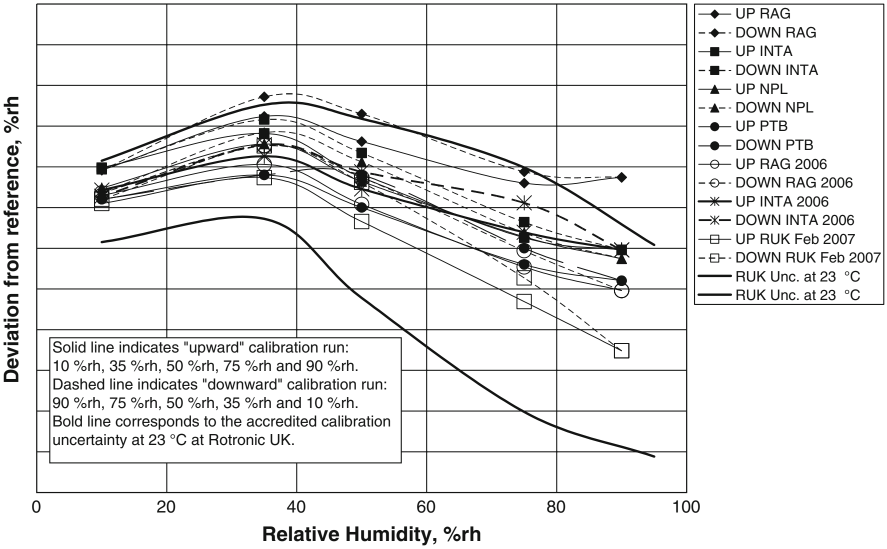

Fig. 9 Results for HygroClip IC-1, Serial Number 36702 002, at 50 °C. Each division of the *y*-axis corresponds to 0.5 %rh

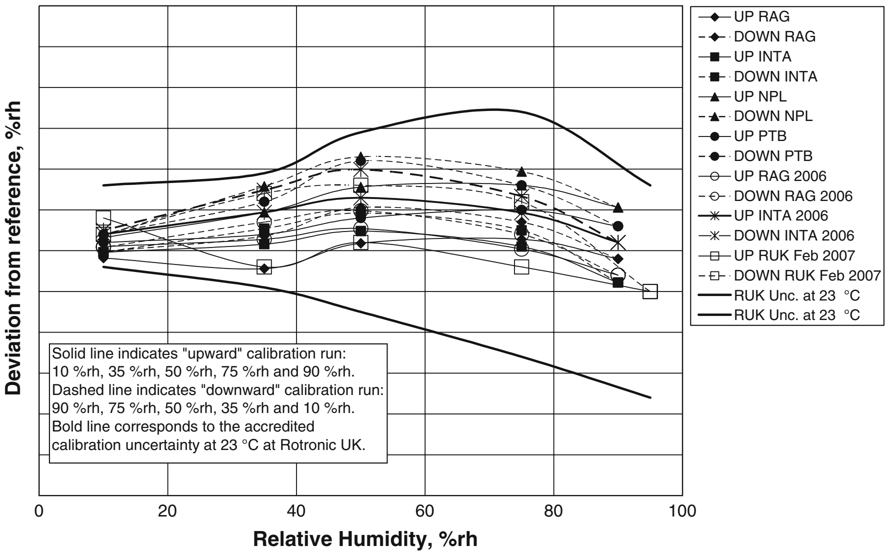

Fig. 10 Results for HygroClip S3, Serial Number 35197 036, at 23 °C. Each division on the *y*-axis corresponds to 0.5 %rh

instruments in group 2 (one of each model) that underwent the full cycling have been used to compare the results and generate tables of degrees of equivalence using measurements in defined directions.

Figures 9–11 show the full results of models IC-1 (002), S3, and S, at the temperatures of 50 °C, 23 °C (including RUK salt-solution values), and 5 °C, respectively. The

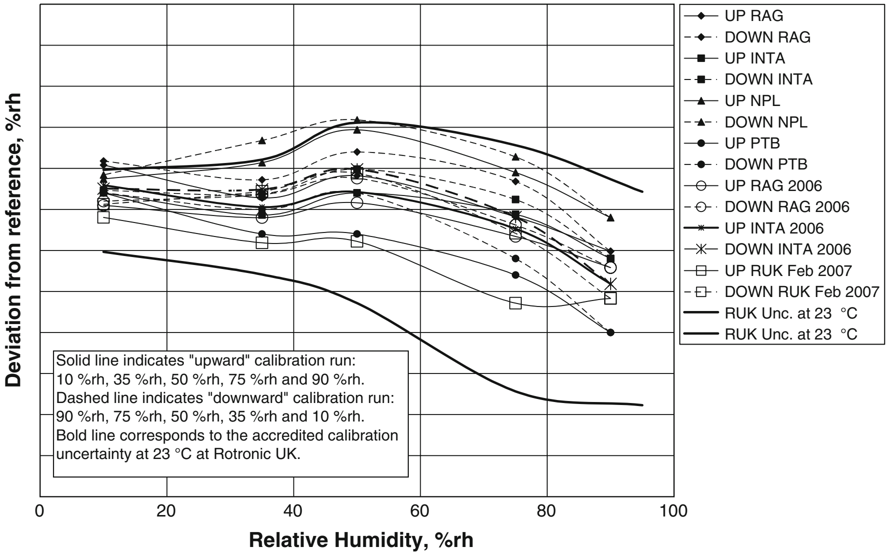

Fig. 11 Results for HygroClip S, Serial Number 37251 187, at 5 °C. Each division on the *y*-axis corresponds to 0.5 %rh

solid curves in each figure show the assigned expanded uncertainty of measurement ($k = 2$) of Rotronic UK at 23 °C for reference. The effects of hysteresis can be clearly seen in all cases.

The degrees of equivalence for the RH results, calculated at the middle of the range at 50 %rh, at the three air temperatures are shown in Tables 3–5, using the results obtained with Models IC-1 (002), S3, and S, for 50 °C (final 50 %rh), 23 °C (initial 50 %rh), and 5 °C (final 50 %rh), respectively. These results are consistent with the limitations inherent to RH capacitive sensors, but have been chosen to minimize the effects of long-term stability and reproducibility. The results tabulated assume that the results of all the laboratories are uncorrelated—which is not strictly true for the Rotronic UK data when comparing results with NPL (using dew-point hygrometer and thermometers) or with Rotronic AG (unsaturated salt solutions). However, the degree of equivalence, including the effects of drift as established by the repeat measurements at INTA, allows direct comparison of the Rotronic UK data with either INTA or PTB. However, an uncorrelated method of showing equivalence of the Rotronic UK data can be achieved if we take INTA as the pilot laboratory, add a component to the uncertainty quoted by the pilot laboratory to take into account the drift of the comparison instruments, and then compare the results using the well-known formula:

$$E_{\mathrm{n}} = \frac{x - X}{\sqrt{U_{\mathrm{lab}}^2 + U_{\mathrm{ref}}^2}}, \quad (1)$$

where E_{n} is the normalized error, U_{lab} the uncertainty of Rotronic UK's result, and U_{ref} is the uncertainty of the pilot laboratory's result. X and x are the pilot laboratory

Table 3 Bilateral equivalence [9,10] between laboratories for instrument HygroClip IC-1 Serial Number 36702 002, at 50 °C and the final 50 %rh calibration point in the run, in terms of pair differences shown with their assigned expanded uncertainties at the 95 % confidence level in terms of %rh

	Initial Rotronic AG	Initial INTA	NPL	PTB	Rotronic AG	INTA	Rotronic UK
Initial Rotronic AG	–	0.17 ± 1.5	0.44 ± 1.5	0.82 ± 1.1	0.54 ± 1.4	0.61 ± 1.5	0.85 ± 1.5
Initial INTA	−0.17 ± 1.5	–	0.27 ± 1.6	0.65 ± 1.2	0.37 ± 1.5	0.44 ± 1.5	0.68 ± 1.6
NPL	−0.44 ± 1.5	−0.27 ± 1.6	–	0.38 ± 1.21	0.10 ± 1.5	0.17 ± 1.6	0.41 ± 1.6
PTB	−0.82 ± 1.1	−0.65 ± 1.2	−0.38 ± 1.2	–	−0.28 ± 1.1	−0.21 ± 1.2	0.03 ± 1.2
Rotronic AG	−0.54 ± 1.4	−0.37 ± 1.5	−0.10 ± 1.5	0.28 ± 1.1	–	0.07 ± 1.5	0.31 ± 1.5
INTA	−0.61 ± 1.5	−0.44 ± 1.6	−0.17 ± 1.6	0.21 ± 1.2	−0.07 ± 1.49	–	0.24 ± 1.6
Rotronic UK	−0.85 ± 1.5	−0.68 ± 1.6	−0.41 ± 1.6	−0.03 ± 1.2	−0.31 ± 1.5	−0.24 ± 1.6	–

Table 4 Bilateral equivalence between laboratories for instrument HygroClip S3 Serial Number 35197 036, from the initial 23 °C run and the initial 50 %rh calibration point in that run, in terms of pair differences shown with their assigned expanded uncertainties at the 95 % confidence level in terms of %rh

	Initial Rotronic AG	Initial INTA	NPL	PTB	Rotronic AG	INTA	Rotronic UK
Initial Rotronic AG	–	−0.11 ± 1.2	−0.68 ± 1.2	−0.29 ± 0.64	−0.17 ± 0.57	−0.51 ± 1.2	−0.19 ± 1.2
Initial INTA	0.11 ± 1.2	–	−0.57 ± 1.6	−0.18 ± 1.2	−0.06 ± 1.2	−0.40 ± 1.6	−0.08 ± 1.6
NPL	0.68 ± 1.2	0.57 ± 1.6	–	0.39 ± 1.2	0.51 ± 1.2	0.17 ± 1.6	0.49 ± 1.6
PTB	0.29 ± 0.64	0.18 ± 1.2	−0.39 ± 1.2	–	0.12 ± 0.64	−0.22 ± 1.2	0.10 ± 1.2
Rotronic AG	0.17 ± 0.57	0.06 ± 1.2	−0.51 ± 1.2	−0.12 ± 0.6	–	−0.34 ± 1.2	−0.02 ± 1.2
INTA	0.51 ± 1.2	0.40 ± 1.6	−0.17 ± 1.6	0.22 ± 1.2	0.34 ± 1.2	–	0.32 ± 1.6
Rotronic UK	0.19 ± 1.2	0.08 ± 1.6	−0.49 ± 1.6	−0.10 ± 1.2	0.02 ± 1.2	−0.32 ± 1.6	–

Table 5 Bilateral equivalence between laboratories for instrument HygroClip S Serial Number 37251 187, at 5 °C and the final 50 %rh calibration point in the run, in terms of pair differences shown with their assigned expanded uncertainties at the 95 % confidence level in terms of %rh

	Initial Rotronic AG	Initial INTA	NPL	PTB	Rotronic AG	INTA	Rotronic UK
Initial Rotronic AG	–	0.11 ± 1.4	−0.50 ± 1.4	0.61 ± 1.0	0.29 ± 1.3	0.19 ± 1.4	0.28 ± 1.4
Initial INTA	−0.11 ± 1.4	–	0.61 ± 1.6	0.50 ± 1.2	0.18 ± 1.4	0.08 ± 1.6	0.17 ± 1.6
NPL	0.50 ± 1.4	0.61 ± 1.6	–	1.11 ± 1.2	0.79 ± 1.4	0.69 ± 1.6	0.78 ± 1.6
PTB	−0.61 ± 1.0	−0.50 ± 1.2	−1.11 ± 1.2	–	−0.32 ± 1.0	−0.42 ± 1.2	−0.33 ± 1.2
Rotronic AG	−0.29 ± 1.3	−0.18 ± 1.4	−0.79 ± 1.4	0.32 ± 1.0	–	−0.10 ± 1.4	−0.01 ± 1.4
INTA	−0.19 ± 1.4	−0.08 ± 1.6	−0.69 ± 1.6	0.42 ± 1.2	0.10 ± 1.4	–	0.09 ± 1.6
Rotronic UK	−0.28 ± 1.4	−0.17 ± 1.6	−0.78 ± 1.6	0.33 ± 1.2	0.01 ± 1.4	−0.09 ± 1.6	–

and test laboratory deviations from the reference, respectively. Laboratories showing good equivalence with each other expect values in the range $-1 < E_\mathrm{n} < 1$ to be returned. In every case and for both groups of instruments, the E_n values calculated when the Rotronic UK data were compared with the INTA data were in the range $-0.75 < E_\mathrm{n} < 0.60$.

4 Conclusions

Six RH and temperature probes were calibrated over a period of 2 years at three European NMIs and the two accredited laboratories of the manufacturer. The obtained RH results show that the instruments selected are suitable for this type of comparison, if due regard is made for the hysteresis of the instruments. The characterization of the probes has shown that optimum values of long-term stability and drift can result if measurements are limited to the required range. The equivalence shown between all laboratories taking part in the comparison, using diverse calibration techniques and traceability to several national standards, is consistent with the assigned expanded uncertainties at the 95 % confidence level at all calibration points. The results further reinforce confidence in the work currently being carried out at the manufacturer's Swiss and UK accredited laboratories.

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