Final report

EURAMET 1382 comparison of the realisations of the dew-point temperature in the range from -40 °C to 20 °C

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The Institute of Metrology of Bosnia and Herzegovina (IMBIH) as the NMI, has recently upgraded its hygrometry facilities with the new primary dew-point generator. To assess its performance, interlaboratory comparison was conducted in the scope of the EURAMET (project 1382) between the IMBIH and the LMK. The comparison range was from -40 °C to 20 °C. To assure traceability to SI, LMK provided the link to the BIPM K6 key intercomparison through the EURAMET.T-K6 (EURAMET Project no. 621) [1,2]. The results of the comparison show a good agreement in the whole dew/frost point range from -40 °C to 20 °C. By linking the results to the EURAMET.T-K6, the deviation from the EURAMET.T-K6 reference value ranges from -0.012 °C to 0.078 °C with expanded uncertainties from 0.091 °C to 0.149 °C.

The technical protocol of the comparison has been drawn up by the LMK in consultation with the IMBIH (see Appendix A). The protocol is based on the one from the the EURAMET.T-K6 and it follows the guidelines established by the BIPM and EURAMET, which are based on best practice in the use of dew/frost-point hygrometers and the experience gained from the regional comparisons over the years.

The drift of the transfer standard

For the transfer standard a dew-point sensor with a 20 years LMK calibration history was selected. It is manufactured by Omega Engineering, RHB-2 chilled mirror hygrometer with serial number 0880196. To assess the drift of the transfer standard, measurements were first performed at LMK, proceeded by IMBIH and finally again at the LMK. They were carried out at nominal frost-point temperatures of -40 °C, -20 °C and -10 °C; and at nominal dew-point temperatures of +7 °C and +20 °C, respectively, in rising order of dew/frost-point temperature. Two set of measurements were carried out (reproduced) in each point to reduce the effect of any irreproducibility of the transfer standard. The first set of measurements at the LMK were performed by comparison of transfer standard against the reference precision chilled mirror hygrometer MBW 373LXHX, which was previously calibrated against the LMK primary dew-point generator LMK-PGR-01. The second and final sets of measurements at the LMK were performed directly against the LMK primary generator LMK-PGR-01 [2]. The latter allowed us to provide the link to the EURAMET.T-K6.

The uncertainty due to drift of the transfer standard, $u_{\mathrm{drift}}(E_{\mathrm{LMK1}} - E'_{\mathrm{LMK2}})$, was calculated for the whole comparison range according to Eq.1. It was obtained by observing the deviation of the transfer standard from the values measured by the MBW 373LXHX. Namely, this sensor was used also in the final sets of measurements at the LMK – LMK2, while it was calibrated in parallel with the transfer standard against the primary dew-point generator.

$$u_{\mathrm{drift}}(E_{\mathrm{LMK1}} - E'_{\mathrm{LMK2}}) = \sqrt{u^2_{\mathrm{ts,LMK1}} + u^2_{\mathrm{ts,LMK2}} - 2 \cdot r(E_{\mathrm{LMK1}}, E'_{\mathrm{LMK2}}) \cdot u_{ts,LMK1} \cdot u_{ts,LMK2}} \quad (1)$$

$u_{\mathrm{ts,LMK1}}$ and $u_{\mathrm{ts,LMK2}}$ represent the uncertainties of the deviation of the transfer standard, when compared/calibrated against the MBW 373LXHX in the both set of measurements at the LMK, E_{LMK1} and E'_{LMK2}, respectively. Both these uncertainties therefore include the uncertainty of the reference value (measured by MBW 373LXHX) and the contribution of the unit under calibration (UUC), the transfer standard. $r(E_{\mathrm{LMK1}}, E'_{\mathrm{LMK2}})$ represents correlation coefficient of the two sets of deviations obtained at first and second LMK measurements, E_{LMK1} and E'_{LMK2}, respectively, for the whole range of dew/frost point, t_{dp}.

Based on the E_{LMK1} and E'_{LMK2} and the uncertainty values $u_{\mathrm{ts,LMK1}}$ and $u_{\mathrm{ts,LMK2}}$, which were estimated at 0.088 °C and 0.071 °C, respectively, the uncertainty of the drift $u_{\mathrm{drift}}(E_{\mathrm{LMK1}} - E'_{\mathrm{LMK2}})$ amounted to 0.026 °C.

The results of the comparison

Both labs provided the average deviation of the transfer standard indication from its reference for the set of two repeated measurements – the results of the comparison, E_{LMK} and E_{IMBIH}, respectively. The uncertainty of each result was calculated according to Eq.2, by combining the uncertainty of the applied dew/frost point value, $u_{\mathrm{ref,LMK}}$ and $u_{\mathrm{ref,IMBIH}}$; then the uncertainty due to

the transfer standard, $u_{\text{uut,LMK}}$ and $u_{\text{uut,IMBIH}}$ and the uncertainty due to the drift of the transfer standard, u_{drift} (see above).

$$u(E_{\text{lab}}) = \sqrt{u^2_{\text{ref,lab}} + u^2_{\text{uut,lab}} + u^2_{\text{drift}}(E_{\text{LMK1}} - E'_{\text{LMK2}})}\,, \tag{2}$$

where index *lab* is to be substituted for an individual laboratory designation. A summary of the comparison results is shown in the Fig.1 and in the Table 1 below.

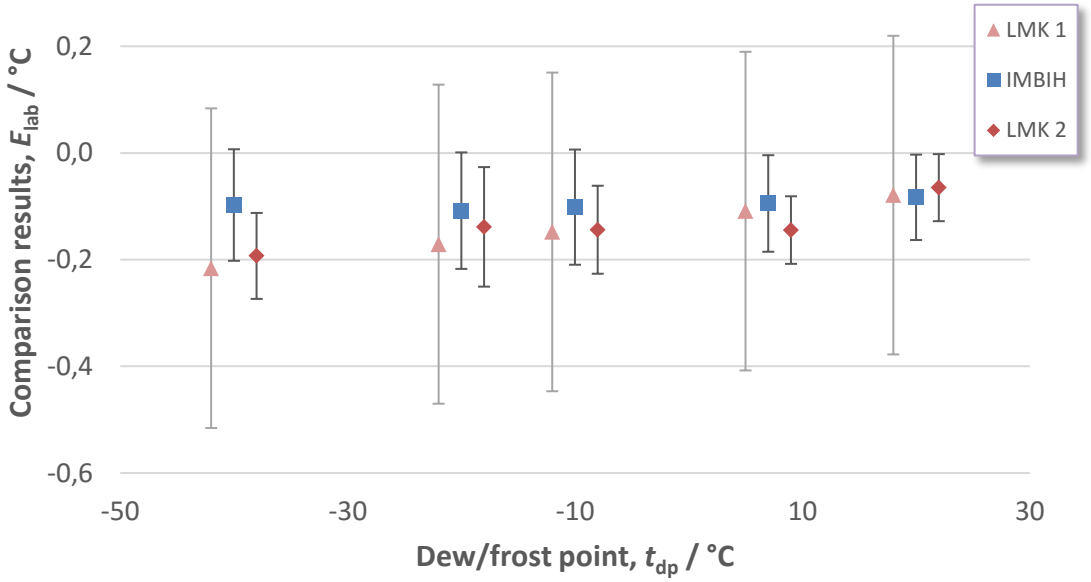

Figure 1. Results of the comparison, together with the assigned uncertainties

Bilateral equivalence

A bilateral equivalence between LMK and IMBIH, $D_{\text{LMK-IMBIH}}$, and its standard uncertainty, $u(D)$ was established according to Eq.3 and Eq.4 [3].

$$D_{\text{LMK-IMBIH}} = E_{\text{LMK}} - E_{\text{IMBIH}} + \delta_{\text{drift}}\,, \tag{3}$$

$$u(D) = \sqrt{u^2_{\text{ref,LMK}} + u^2_{\text{uut,LMK}} + u^2_{\text{ref,IMBIH}} + u^2_{\text{uut,IMBIH}} + u^2(\delta_{\text{drift}})}\,,$$

$$u^2(\delta_{\text{drift}}) = u^2_{\text{drift}}(E_{\text{LMK1}} - E'_{\text{LMK2}})\,, \tag{4}$$

where the mean correction due to the drift of the transfer standard, δ_{drift}, is estimated to be zero, but its standard uncertainty is $u_{\text{drift}}(E_{\text{LMK1}} - E'_{\text{LMK2}})$, as estimated above.

The summary of the results is shown below in the Table 1.

It can be observed that in the whole range $|D| < U(D)$, which shows a good agreement of the results. $U(D)$ represents an expanded uncertainty with coverage factor k=2.

Link to the EURAMET.T-K6 Comparison

IMBIH results can be linked to the EURAMET.T-K6 comparison reference value E_{K6}, through the LMK results in that comparison, $E_{LMK,K6}$ [1]. As explained above, LMK used the same reference as in the time of the EURAMET.T-K6 without any adaptation on the primary generator in this range. Therefore, the difference between the reference value E_{K6} and the IMBIH results, E_{IMBIH} can be calculated by Eq. 5 [3].

$$E_{\mathrm{IMBIH,K6}} = E_{\mathrm{LMK,K6}} - D_{\mathrm{LMK\text{-}IMBIH}} \tag{5}$$

The corresponding uncertainty is calculated according to Eq.6.

$$u\left(E_{\mathrm{IMBIH,K6}}\right) = \sqrt{u^2\left(E_{\mathrm{LMK,K6}}\right) + u^2\left(D_{\mathrm{LMK\text{-}IMBIH}}\right)} \tag{6}$$

Because IMBIH primary generator is limited to frost point -40 °C, some of the nominal points of this comparison are different from the points EURAMET.T-K6. The range of this comparison does not, however go beyond the range of the EURAMET.T-K6. For this reason, the LMK results, $E_{LMK,K6}$ are linearly interpolated between the neighbouring points, while the uncertainties are taken conservatively as the maximum value in the specific subrange.

The summary of the results is shown below in the Table 1. It can be seen that the deviation from the EURAMET.T-K6 reference value ranges from -0.012 °C to 0.078 °C with expanded uncertainties from 0.091 °C to 0.149 °C.

Table 1. The summary of the results of the interlaboratory comparison

t_{dp} /°C	lab	E_{lab} /°C	$U(E_{lab})$/°C	$D_{LMK-IMBIH}$/°C	$U(D)$/°C	$E_{IMBIH,K6}$ /°C	$U(E_{IMBIH,K6})$/°C
-40	IMBIH	-0.098	0.105				
	LMK	-0.193	0.081	-0.095	0.121	0.078	0.125
-20	IMBIH	-0.108	0.109				
	LMK	-0.139	0.112	-0.030	0.148	0.029	0.149
-10	IMBIH	-0.102	0.108				
	LMK	-0.144	0.082	-0.042	0.125	0.044	0.127
7	IMBIH	-0.095	0.090				
	LMK	-0.144	0.063	-0.050	0.097	0.060	0.100
20	IMBIH	-0.083	0.080				
	LMK	-0.065	0.063	0.018	0.088	-0.012	0.091

References

1. M. Heinonen, et al., Investigation of the Equivalence of National Dew-Point Temperature Realizations in the −50 °C to + 20 °C Range, Vol.33, No. 8, pp 1422-1437, 2012
2. D. Hudoklin, J.Bojkovski, J.Nielsen, J.Drnovsek, Design and Validation of a New Primary Standard for Calibration of the Top-end humidity sensors, Measurement 41(2008)
3. M. Heinonen, D. Zvizdic, D. Sestan, Intercomparison of the Dew-Point Temperature Realizations at LPM and MIKES in the Range from −70◦C to+20◦C, Int.J. Thermophysics, Vol. 33, pp. 1451-1457, 2012

Appendix A – The technical protocol of the comparison

P1382 EURAMET Intercomparison of the realisations of the dew-point temperature in the range from -40 °C to 20 °C

Technical protocol

CONTENTS

1 INTRODUCTION 7
2 ORGANIZATION 7
2.1 Participants 7
2.2 Method of comparison 7
2.3 Handling of artefacts 8
3 DESCRIPTION OF THE TRAVELLING STANDARD 8
3.1 Travelling standard 8
4 MEASUREMENT INSTRUCTIONS 9
4.1 Measurement process 9
4.2 Data collection 10
5 REPORTING OF MEASUREMENT RESULTS 10
6 UNCERTAINTY OF MEASUREMENT 12
7 DETERMINATION OF THE COMPARISON REFERENCE VALUE 12
APPENDIX 1. DETAILS OF PARTICIPATING INSTITUTES 13

1 INTRODUCTION

In October 2015, it was agreed to organise a EURAMET Inter-comparison of the realisations of the dew-point temperature in the range from -40 °C to 20 °C.

This technical protocol has been drawn up by the pilot in consultation with the nominated participants listed in Section 2. The protocol is based on the protocol from the key-comparison Euromet P621.

The procedures outlined in this document cover the technical procedure to be followed during measurement of the travelling standard. The procedure, which follows the guidelines established by the BIPM and EURAMET, is based on current best practice in the use of dew/frost-point hygrometers and takes account of the experience gained from the regional comparisons over the years.

This comparison is aimed at establishing the degree of equivalence between realisations of local scales of dew/frost-point temperature of humid gas, in the range from -40 °C to +20 °C, among the participating national metrology institutes.

ORGANIZATION

Participants

This is a bilateral intercomparison between the University of Ljubljana, Faculty of Electrical Engineering, Laboratroy of Metrology (LMK) as the coordinator and the pilot, and the Institute of Metrology of Bosnia and Herzegovina (IMBIH). Please, see the Appendix 1 for contact details.

By their declared intention to participate in this comparison, the laboratories accept the general instructions and the technical protocol written down in this document and commit themselves to follow strictly the procedures of this protocol as well as the version of the "Guidelines for Key Comparisons" in effect at the time of the initiation of the comparison.

Once the protocol and list of participants have been approved, no change to the protocol or list of participants may be made without prior agreement of all participants.

All participants must be able to submit an uncertainty budget of their humidity standard.

Method of comparison

This is a comparison of the realisations of local scales of dew/frost-point temperature at the participating national institutes.

The comparison will be made by calibration of a travelling standard lent by the LMK. The travelling standard will measure dew/frost-point temperature of a sample of moist gas produced by a participant's standard generator.

Measurements will start in the pilot laboratory. The other participant will then perform comparison measurements at the dew/frost-point temperatures required and then return the travelling standard to the pilot of the loop to carry out final measurements to monitor drift.

All results are to be communicated to the pilot within eight weeks of the completion of the measurements by a laboratory.

In case of serious difficulty with customs, or other delays which might over-run the time period of the ATA Carnet, the pilot may request the instruments be returned to the holder of the ATA Carnet (LMK).

Handling of artefacts

The travelling standard should be examined immediately upon receipt at the laboratory. The participants are expected to follow all instructions in the operator's manual provided by the instrument manufacturers for proper unpacking, subsequent packing and shipping to the next participant. During packing and unpacking, all participants should check the contents with the packing list.

The travelling standard should only be handled by authorized persons and stored in such a way as to prevent damage.

2.4 Transport of artefacts

The travelling standard is hand carried in a container, which is sufficiently robust to ensure safe transportation. The artefact will be accompanied by a suitable customs ATA Carnet. Care should be taken with the timing of the ATA Carnet, which only lasts for one year.

DESCRIPTION OF THE TRAVELLING STANDARD

Travelling standard

LMK lends a travelling standard per loop for the comparison. The instrument is a precision commercially available chilled-mirror type dew-point hygrometer. The travelling standard has a long and stable calibration history in the LMK.

The details of the travelling standard:

Model:	RHB-2, RHB-S (sensor head)
Weight (in packing case):	7 kg
Manufacturer:	Omega Engineering (General Eastern)
Owner:	LMK
Electrical supply:	230 V / 50 Hz
Tube connectors:	Swagelok ¼”
Communication:	RS232
Accessories:	Connection cable to the sensor head; RS232 comm. cable
Serial number:	0880196

MEASUREMENT INSTRUCTIONS

Measurement process

The participants should refer to the operating manuals for instructions and precautions for using the travelling standard. Participants may perform any initial checks of the operation of the hygrometer that would be performed for a normal calibration. In the case of an unexpected instrument failure at a participant institute, the pilot institute should be informed in order to revise the time schedule, if necessary, as early as possible.

Sample gas generated by a participant's standard generator, is introduced into the inlet of a travelling standard hygrometer through a stainless steel tube terminating with a Swagelok ¼ inch fitting.

Measurements are carried out at nominal dew-point temperatures of +20 °C and +7 °C; and nominal frost-point temperatures of –10 °C, -20 °C and –40 °C. In the range below 0 °C, a homogenous ice layer should cover the mirror and participants should report the applied condition in terms of frost-point temperature. The phase of condensate apparent on the mirror of the travelling standard should also be reported.

Measurements should be done in rising order of dew/frost-point temperature.

Two set of measurements are carried out (reproduced) to reduce the effect of any irreproducibility of the travelling standard. The condensate should be cleared and re-formed for each value or repetition of dew/frost-point temperature.

The values of dew/frost-point temperature applied to the travelling standard should be within ±2 °C of the five agreed nominal values for the comparison, and ideally closer than this. Deviations greater than this may increase the uncertainty in the comparison, for a particular result.

Before any humidity measurements, initial actions should be taken:

- Read the manual “Operating Instructions” delivered by the manufacturer.

- Clean the mirror surface using cotton tips with distilled or de-ionised water preceded by initial cleaning with alcohol if necessary.

- Clean the mirror in the standard and initiate Pacer.

- Set the flow rate of sample gas between 0.5 l/min and 1 l/min.

Participants should avoid lengthy additional measurements, except those necessary to give confidence in the results of this comparison.

The travelling standard used in this comparison must not be modified, adjusted, or used for any purpose other than described in this document, nor given to any party other than the participants in the comparison.
The Pilot will make an assessment of any drift in the travelling standard during the comparison, based on measurements at the pilot laboratory at the beginning and end of the comparison period. If drift is found, this will be taken into account in the final analysis of the comparison results as an additional source of uncertainty of the reference value.

If poor performance or failure of a travelling standard is detected, the pilot of the loop will propose a course of action, subject to agreement of the participants.

Data collection

Each measured value (incl. its experimental standard uncertainty) is obtained calculating the mean and standard deviation of at least 10 readings of the resistance of the PRT recorded during 10 to 20 minutes.

Participants may apply their own criteria of stability for acceptance of measurements.

Dew/frost-point temperatures of the travelling standard should be recorded through RS232 port in order to obtain resolution better than 0,1 °C. The mean and standard deviation a set of at least 10 readings, taken over the same period as the dew/frost point measurements should be reported.

Values reported for dew/frost-point temperatures produced by a participant's standard generator should be the value applied to the instruments, after any allowances for pressure and temperature differences between the point of realisation (laboratory standard generator) and the point of use (travelling standard).

REPORTING OF MEASUREMENT RESULTS

Participants must report their measurement results within eight weeks of completing their measurements.

The pilot should accumulate data continually and should analyse the results for possible anomalies in the travelling standard. If problems arise, the pilot should consult with the

participant that submitted the data as soon as possible, and certainly before the distribution of Draft A of the report of the comparison.

The parameter to be compared between the laboratories in this comparison is the difference found between the travelling standard and the laboratory dew-point temperature standard. The travelling standard is used simply as comparator.

Participants should report results to the pilot in terms of dew/frost-point temperature. The main measurement results comprise:

- values of dew/frost-point applied to the travelling standard, and associated standard uncertainty;
- values measured by a travelling standard (and associated uncertainties derived from standard deviation of the set of readings);
- values of difference between applied dew/frost point and measured dew/frost point.

A template for reporting of results will be made available to participants in electronic form as an Excel spreadsheet. Use of this format, including calculations of means and differences, allows participants to see clearly the values and uncertainties of the parameters they are submitting for comparison.

From the data measured by each participant, results will be analysed in terms of differences between applied (realised) and measured dew-point temperatures.

The participants should report the conditions of realisation and measurement, as background information to support the main results. These conditions may include, pressure and temperature in saturator, pressure difference between saturator and travelling standard, measurement traceability and other items. The conditions of measurement should be reported in the same template as for reporting of results.

Participants should provide a description of the operation of their dew/frost-point facilities used in the comparison.

Participants should also provide an example plot of equilibrium condition (frost-point temperature versus time) at a nominal value of -40 °C over a period of at least one hour.

Any information obtained relating to the use of any results obtained by a participant during the course of the comparison shall be sent only to the pilot laboratory and as quickly as possible. No communication whatsoever regarding any details of the measurement other than the general conditions described in this protocol shall occur between the participants or any party external to the comparison without the written consent of the coordinator. This is to ensure that no bias from whatever accidental means can occur. These constraints on communication apply until the circulation of draft A of the report of the comparisons.

UNCERTAINTY OF MEASUREMENT

The uncertainty of the comparison results will be derived from:

- the quoted uncertainty of the dew/frost-point realisation (applied dew/frost point temperature)
- the estimated uncertainty relating to the short-term stability of the travelling standard at the time of measurement
- the estimated uncertainty due to any drift of the travelling standard over the period of the comparison (estimated by the pilot)
- the estimated uncertainty in mean values due to dispersion of repeated results (reflecting the combined reproducibility of laboratory standard and travelling standard)
- the estimated uncertainty due to non-linearity of the travelling standard in any case where measurements are significantly away from the agreed nominal value
- the estimated covariance between applied (laboratory standard) and measured (travelling standard) values of dew/frost-point temperature (if found significant)
- any other components of uncertainty that are thought to be significant

Participants are required to submit detailed analyses of uncertainty for their dew-point standard.

Uncertainty analysis should be according to the approach given in the ISO Guide to the Expression of Uncertainty of Measurement. A list of the all significant components of the uncertainty budget should be evaluated, and should support the quoted uncertainties.

Uncertainties shall be reported together with other measurement results and conditions in the same report (according to a template). Participants may add to the template any additional uncertainties they consider relevant.

The pilots will review the uncertainty budgets for consistency among participants.

The uncertainty budget stated by the participating laboratory should be referenced to an internal report and/or a published article.

DETERMINATION OF THE COMPARISON REFERENCE VALUE

The outputs of the comparison are expected to be:

- results of individual participant for comparison of the hygrometers against their dew point reference at each measured value, estimated standard uncertainty of each result and estimated standard uncertainty of comparison process (e.g. effect of long-term stability and non-linearity of the travelling standard), if necessary;

- a comparison reference value (RV) for each nominal value of dew/frost point temperature in the comparison. The RV will be provided by the LMK.

- estimates of equivalence of IMBIH value to the key comparison reference value (KCRV) of the EUROMET.T-K6 (EURAMET 621 project) key intercomparison and thus giving linkage between comparisons via MIRS/UL-FE/LMK. This might be expressed as the Degree of Equivalence (DOE) given as the difference and its uncertainty ($\Delta \pm U$).

In the field of dew-point standard, the RV does not have any absolute significance. It is calculated only for purposes such as the presentation and inter-relation of comparison data for the MRA.

The pilot will make an assessment of any drift in the travelling standard during the comparison. The assessment will be based on initial and final measurements done by the Pilots. If drift is found, it will be taken into account in the final analysis of the comparison results. If the drift is small compared to the uncertainty values reported by the participants, an estimate for the drift may be set to zero with a standard uncertainty calculated according to the ISO Guide. In a case of a significant drift, the effect is taken into account by assigning a time-dependent value to RV, or by other suitable method so that the estimates of equivalence can be meaningfully calculated between results taken at different times.

If a travelling standard fails or performs poorly during the comparison, the coordinator will propose a course of action, subject to agreement of the participants.

APPENDIX 1. DETAILS OF PARTICIPATING INSTITUTES

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