SIM.T-K6.7

COMPARISON OF HUMIDITY STANDARDS

Instituto Nacional de Metrologia, Qualidade e Tecnologia (INMETRO), Brazil
Instituto Nacional de Tecnología Industrial (INTI), Argentina

Dew/Frost-Point Temperature –30 °C to +60 °C

TECHNICAL PROTOCOL

April 2017

Contents

1. INTRODUCTION

1.1. Under the Mutual Recognition Arrangement (MRA)[1] the metrological equivalence of national measurement standards will be determined by a set of key comparisons chosen and organized by the Consultative Committees of the CIPM working closely with the Regional Metrology Organizations (RMOs).

1.2. At its 20th meeting in April 2000, the Consultative Committee for Thermometry, CCT, considered a Key Comparison on humidity as imperative for the related laboratories. This document is based on a technical protocol drawn up by the members of Working Group on Humidity Measurements (CCT-WG-Hu).

1.3. It is appropriate to have a comparison of humidity standards between *Instituto Nacional de Metrologia, Qualidade e Tecnologia* (INMETRO), Brazil, and *Instituto Nacional de Tecnología Industrial* (INTI), Argentina.

1.4. The two participants indicated above have prepared this technical protocol.

1.5. The procedures outlined in this document cover the technical procedure to be followed during measurement of a transfer standard. The procedure, which follows the guidelines established by the BIPM[2], is based on current best practice in the use of dew/frost-point hygrometer and takes account of the experience gained from the research and calibration activities of the participants over the years.

1.6. This comparison is aimed at checking the degree of equivalence between realisations of local scales of dew/frost-point temperature of humid air established in a previous comparison among the participating National Metrology Institutes (NMIs)[3], and expand it to a wider range (from -30 °C to +60 °C).

1.7. INTI's results for the dew-point temperatures 1 ºC and 20 ºC will be linked to CCT-K6 key comparison reference value by means of a previous comparison between INMETRO and the National Institute of Standards and Technology (NIST, USA). Detailed information about it can be found in chapter 7.

2. ORGANIZATION

2.1. **Participants**

2.1.1. Details of mailing and electronic addresses are given in Appendix 1. The participating institutes are:

- Instituto Nacional de Metrologia, Qualidade e Tecnologia (INMETRO) – Brazil
- Instituto Nacional de Tecnología Industrial (INTI) – Argentina

2.1.2. INMETRO is the Pilot of the comparison, taking main responsibility for running the comparison.

[1] MRA, Mutual Recognition Arrangement, BIPM, 1999.
[2] CIPM MRA-D-05, Version 1.6, March 2016.
[3] J. D. Brionizio and J. G. Skabar. *Final Report on SIM.T-K6.4: Comparison of INMETRO and INTI Humidity Standards*. Metrologia, Vol. 50, Tech. Suppl., 03010 (2013)

2.1.3. 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.

2.1.4. 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.

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

2.2. **Method of Comparison**

2.2.1. The comparison will be made by means of the calibration of a travelling transfer standard. The transfer standard will independently measure dew/frost-point temperature of a sample of moist air produced by a participant's standard system using the same measuring process.

2.2.2. Circulation scheme

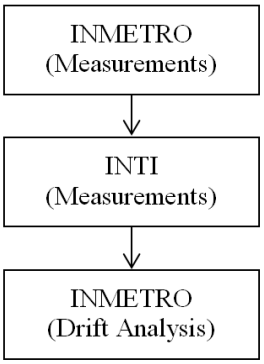

Figure 1 – Circulation scheme of the comparison

2.3. **Handling of Artefact**

2.3.1. The artefact shall be examined before the start of measurements. The participants are expected to follow all instructions in the operator's manual provided by the instrument manufacturer for proper unpacking, subsequent packing and operation. During packing and unpacking, the participants shall check the contents with the packing list including the operator's manual.

2.3.2. The transfer standard must only be handled by authorized persons and stored in such a way as to prevent damage.

2.3.3. During operation of the transfer standard, if there is any unusual occurrence, e.g., loss of heating or cooling control, the Pilot laboratory shall be notified immediately before proceeding.

2.4. **Transport of Artefact**

2.4.1. The transfer standard will be hand-carried from INMETRO to INTI by an INMETRO’s technician, which will be at INTI while comparison measurements are conducted at the dew/frost-point temperatures required. After that, the transfer standard will be hand-

carried from INTI to INMETRO by the INMETRO's technician. Each participant shall take actions in order to guarantee the exit and entrance of the transfer standard in its country.

2.5. **Shipping Costs**

2.5.1. INTI will be responsible for the travelling costs and daily allowances of an INMETRO's technician who will hand-carry the transfer standard. Each institute will be responsible for the customs charges. INMETRO will be responsible for the insurance of the transfer standard, which shall be sufficient to cover the costs of the item and any damages that may occur.

2.6. **Timetable**

Table 1 – Timetable of the comparison

Activity	Start Month	Provisional Date
Submission of a technical protocol to participants for unanimous approval	May 2016	
Submission of revised technical protocol to SIM/ WG3 (thermometry WG) for approval.		December 2016
Completion of measurements at INMETRO		March 2017
Travelling standard hand-carried to INTI		March 2017
Completion of measurements at INTI		April 2017
Measurements at INMETRO to check the transfer standard stability (if necessary)		May 2017
Report A ready		December 2017
Deadline for comments on report A		February 2018
Draft B ready and submitted to SIM/WG3		March 2018
Paper publication		December 2018

3. DESCRIPTION OF THE TRANSFER STANDARD

3.1. **Artefact**

3.1.1. The travelling standard selected for the comparison is a state-of-the-art dew-point hygrometer, chilled-mirror type, commercially available. It has proven to be robust with known performance characteristics such as repeatability and transportability.

3.1.2. Details of travelling standard:

Table 2 – Details of the transfer standard

Manufacturer:	Michell Instruments Ltd., UK
Model:	Optidew Vision
Serial Number:	118931 (display) / 118849 (sensor)
Size:	260 mm (d) x 290 mm (w) x 120 mm (h)
Weight:	2.5 kg
Size in packing case:	300 mm (d) x 460 mm (w) x 200 mm (h)
Owner:	INMETRO, Brazil
Electrical supply:	90-264 V / 47-440 Hz
Approximate value for insurance and customs declaration:	US$ 25,000

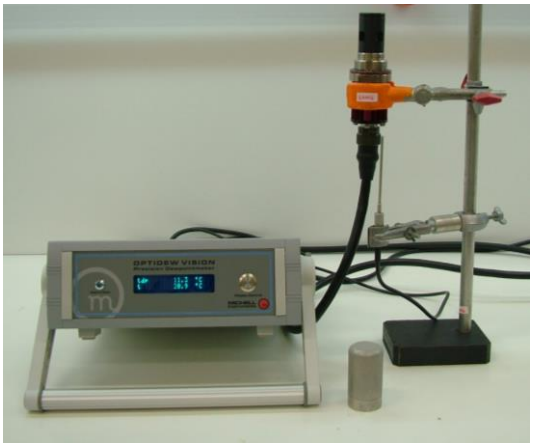

Figure 2 – Travelling standard

4. MEASUREMENT INSTRUCTIONS

4.1. **Measurement Process**

4.1.1. The participants shall refer to the operating manual 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 shall be informed in order to revise the time schedule, if necessary, as early as possible.

4.1.2. A total of six humidity points are used for the comparison. Four dew-point temperatures at nominal values of 1 °C, 20 °C, 40 °C and 60 °C and two frost-point temperatures at nominal values of –30 °C and –20 °C. The value of 1 °C nominally represents 0 °C, while avoiding any complication due to phase change between water and ice.

4.1.3. For the frost/dew-point temperature values of –30 °C, –20 °C and 1 ºC, the gas sample generated by a participant's standard/working generator can be introduced into the inlet of the sensor housing of the travelling standard hygrometer (through a stainless steel tube, or by means of a Teflon hose, terminating with a 6 mm Swagelok fitting), or the sensor can be placed directly in the chamber of the generating system without its housing. For the dew-point temperatures values of 20 °C, 40 °C and 60 °C, the sensor of the transfer standard shall be placed directly in the chamber of the generating system without its housing.

4.1.4. When the sensor is placed directly inside of the chamber of the generating system, the temperature of the chamber shall be from 10 ºC to 30 ºC above the dew-point temperature.

4.1.5. At –30 °C and –20 °C, participants shall report the applied condition in terms of frost-point temperature. The measured condition at these temperatures will be assumed to be with respect to ice, unless otherwise reported.

4.1.6. It is recommended that measurements are performed in rising order of dew/frost point.

4.1.7. The condensate shall be cleared and re-formed for each value or repetition of dew/frost point.

4.1.8. The values of dew/frost point applied to the travelling standard shall be within ±0.5 °C of the six 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.

4.1.9. The conditions for operation of the travelling standard:

(1) Clean the mirror surface using cotton tips with distilled or de-ionised water. This may be preceded by initial cleaning with alcohol if necessary;
(2) When the sensor is placed in the sampling block, set the indicated flow rate of the gas sample from 0.1 l/min to 2 l/min, according to the hygrometer's specifications. When the sensor is inserted directly inside of the generator's chamber, do not place it near any input or output gas flow. The sensor shall be placed approximately in the middle of the chamber. The maximum gas velocity for direct insertion is 10 m/s, for higher values the sintered guard shall be used;
(3) The dew/frost-point indication of the hygrometer is either read from the hygrometer display or acquired by computer through the instrument's serial port. Each participant must report the measurement way chosen.

4.1.10. Each dew/frost-point temperature shall be separately repeated (reproduced) four times to reduce the effect of any irreproducibility of the travelling standard. For each time, at least 10 readings taken over a period of 10 to 20 minutes shall be acquired.

4.1.11. The transfer 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.

4.1.12. 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 in the end of the comparison period.

4.1.13. If unacceptable performance or failure of the travelling standard is detected, the participants will discuss the situation and agree a course of action.

4.2. **Data Collection**

4.2.1. At each measured value, the mean and standard deviation of multiple readings of the displayed dew/frost-point temperature shall be monitored. Participants may apply their own criteria of stability for acceptance of measurements. When hygrometer is in equilibrium with the gas sample, the standard deviation of a set of the readings, taken over a period of 10 to 20 minutes, is likely to be no more than 0.025 °C approximately.

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

5. REPORTING OF MEASUREMENT RESULTS

5.1. Participants must report their measurement results of four repeated experiments, within six weeks of completing their measurements.

5.2. The participants must not disclose their measurement results to a third party. The participants will exchange their measurement results after all the measurements are completed.

5.3. The parameter to be compared between the two laboratories in this bilateral comparison is the mean difference found between the laboratory humidity standard system and the travelling standard. Note that the values of dew/frost-point temperature reported for the travelling standard are "arbitrary" values calculated from the readings. The travelling standard is used simply as a comparator.

5.4. Participants shall report results to each other 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 of difference between applied dew/frost point and measured dew/frost point.

A provisional template for reporting results is shown in Appendix 3, and can be available to participants in electronic form as an Excel spreadsheet.

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

5.6. Participants shall provide a general description of the operation of their dew/frost points apparatus and humidity generator systems.

5.7. Participants shall also provide an example plot of equilibrium condition at a nominal frost-point temperature of –30 ºC, over a suggested period of at least one hour.

6. UNCERTAINTY OF MEASUREMENT

6.1. The uncertainty of the comparison results will be derived from some or all of:

- the quoted uncertainty of the dew/frost-point realisation (applied dew/frost point) including any uncertainties due to pressure drop or other influences acting between the point of realisation and the point of use (travelling standard);
- 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 a 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 generator and travelling standard);
- the estimated uncertainty due to the resolution of the travelling standard (if found to be significant);
- 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 (generator/system) and measured (travelling standard) values of dew/frost-point (if found to be significant); and
- any other components of uncertainty that are thought to be significant.

6.2. Uncertainty analyses shall be according to the approach given in the Guide to the Expression of Uncertainty of Measurement[4]. A list of the all significant components of the uncertainty budget shall be evaluated, and must support the quoted uncertainties. Evaluations shall be given at a level of one standard uncertainty. Type B estimates of uncertainty may be regarded as having infinite degrees of freedom, or an alternative estimate of the number of degrees of freedom may be made following the methods in the Guide.

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

7. DEGREES OF EQUIVALENCE

7.1. The Degree of Equivalence (DoE) of a measurement standard relative to a key comparison reference value, KCRV, is expressed quantitatively by two terms: its deviation from the KCRV and the expanded uncertainty of this deviation computed at a 95% level of confidence[2].

7.2. The DoE between two measurement standards is expressed quantitatively by two terms: the difference between their respective deviations from the KCRV and the expanded uncertainty of this difference computed at a 95% level of confidence[2].

7.3. NIST/KCRV

The National Institute of Standards and Technology (NIST, USA) participated in the CCT-K6 multilateral key comparison in dew/frost-point temperature, $T_{DP/FP}$, from -50 ºC to +20 ºC[5]. For each nominal $T_{DP/FP}$ a key comparison reference value was calculated. The difference for dew/frost-point temperatures (expressed in ºC) between NIST and KCRV, $D_{NIST/KCRV}$, is defined as:

$$D_{NIST/KCRV} = \left(R_{DP/FP}\right)_{NIST} - \left(R_{DP/FP}\right)_{KCRV} \qquad (1)$$

Where, $(R_{DP/FP})_{NIST}$ and $(R_{DP/FP})_{KCRV}$ are the measured and applied dew/frost-point temperature of NIST and KCRV, respectively.

The combined uncertainty of the difference, $u(D_{NIST/KCRV})$, is defined as:

$$u^2\left(D_{NIST/KCRV}\right) = u^2\left(R_{DP/FP}\right)_{NIST} + u^2\left(R_{DP/FP}\right)_{KCRV} + u^2{}_{DRIFT} \qquad (2)$$

Where, $u(R_{DP/FP})_{NIST}$ and $u(R_{DP/FP})_{KCRV}$ are the uncertainties of the measured and applied dew/frost-point temperature of NIST and KCRV, respectively, and u_{DRIFT} is the uncertainty in the comparison due to the drift of the hygrometers.

The expanded uncertainty of the difference, $U(D_{NIST/KCRV})$, with coverage factor k = 2, which provides a coverage probability of approximately 95% for sufficiently large effective number of degrees of freedom of $u(D_{NIST/KCRV})$, is defined as:

$$U\left(D_{NIST/KCRV}\right) = 2u\left(D_{NIST/KCRV}\right) \qquad (3)$$

[4] Joint Committee for Guides in Metrology (JCGM). *Evaluation of measurement data – Guide to the expression of uncertainty in measurement*. 1st ed. (2008)

[5] S. Bell et al. *Final report to the CCT on key comparison CCT-K6 – Comparison of local realisations of dew-point temperature scales in the range –50 °C to +20 °C*. Metrologia, Vo. 52, Tech. Suppl., 03005 (2015)

The DoE between NIST and KCRV is defined as:

$$\left(D_{NIST/KCRV}, U_{NIST/KCRV}\right) = \left[D_{NIST/KCRV}, ku\left(D_{NIST/KCRV}\right)\right] \qquad (4)$$

The DoE of NIST in the CCT-K6 multilateral key comparison are:

Table 3 – Degree of equivalence between NIST and KCRV

	-50 ºC	-30 ºC	-10 ºC	1 ºC	20 ºC
$D_{NIST/KCRV}$ / ºC	-0.128	-0.072	-0.039	-0.011	-0.006
$U_{NIST/KCRV}$ / ºC	0.030	0.038	0.043	0.060	0.050

7.4. **INMETRO/NIST**

Identified as SIM.T-K6.3, NIST and INMETRO performed a bilateral key comparison of their humidity standards in the dew/frost-point temperature range from -30 ºC to +20 ºC[6]. Some results of this bilateral comparison are linked to the KCRV.

The difference for dew/frost-point temperatures (expressed in ºC) between INMETRO and NIST, $D_{INMETRO/NIST}$, is defined as:

$$D_{INMETRO/NIST} = \left(R_{DP/FP}\right)_{INMETRO} - \left(R_{DP/FP}\right)_{NIST} \qquad (5)$$

Where, $(R_{DP/FP})_{INMETRO}$ and $(R_{DP/FP})_{NIST}$ are the measured dew/frost-point temperatures of INMETRO and NIST, respectively.

The combined uncertainty of the difference, $u(D_{INMETRO/NIST})$, is defined as:

$$u^2\left(D_{INMETRO/NIST}\right) = u^2\left(R_{DP/FP}\right)_{INMETRO} + u^2\left(R_{DP/FP}\right)_{NIST} + u^2{}_{DRIFT} \qquad (6)$$

Where, $u(R_{DP/FP})_{INMETRO}$ and $u(R_{DP/FP})_{NIST}$ are the uncertainties of the measured dew/frost-point temperatures of INMETRO and NIST, respectively, and u_{DRIFT} is the uncertainty in the comparison due to the drift of the transfer standard.

The expanded uncertainty of the difference, $U(D_{INMETRO/NIST})$, with coverage probability of approximately 95% (k = 2) is defined as:

$$U\left(D_{INMETRO/NIST}\right) = 2u\left(D_{INMETRO/NIST}\right) \qquad (7)$$

The DoE of INMETRO in the bilateral key comparison are:

Table 4 – Degree of equivalence between INMETRO and NIST

	-30 ºC	-10 ºC	0 ºC	20 ºC
$D_{INMETRO/NIST}$ / ºC	-0.040	0.083	0.050	0.018
$U_{INMETRO/NIST}$ / ºC	0.20	0.20	0.20	0.20

[6] P. H. Huang, C. W. Meyer, J. D. Brionizio. *Bilateral Key Comparison SIM.T-K6.3 on Humidity Standards in the Dew/Frost-point Temperature Range from –30 °C to 20 °C*. Metrologia, Vol. 52, Tech. Suppl., *03001* (2015)

7.5. **INMETRO/KCRV**

The CCT-K6 comparison was performed at $T_{DP/FP}$ values of 20 °C, 1 °C, −10 °C, −30 °C and −50 °C. The last two values are not considered here since the NIST Hybrid Humidity Generator was not used at those points in CCT-K6. As the CCT-K6 comparison was performed at 1 °C and the comparison between INMETRO and NIST was performed at 0 °C, the participants consider these values acceptably close for linkage and assume that[7]:

$$D_{NIST/KCRV}(1\,°C) = D_{INMETRO/NIST}(0\,°C) \qquad (8)$$

Since INMETRO did not participate in CCT-K6 comparison, Eqs. (1) and (5) may be used to determine $D_{INMETRO/KCRV}$:

$$D_{INMETRO/KCRV} = D_{INMETRO/NIST} + D_{NIST/KCRV} \qquad (9)$$

The expanded uncertainty of the difference, $U(D_{INMETRO/KCRV})$, with coverage probability of approximately 95% (k = 2) is defined as:

$$U^2(D_{INMETRO/KCRV}) = U^2(D_{INMETRO/NIST}) + U^2(D_{NIST/KCRV}) \qquad (10)$$

Combining the results of Tables 3 and 4 and using Eqs. (9) and (10), the values to $D_{INMETRO/KCRV}$ and $U(D_{INMETRO/KCRV})$ are:

Table 5 – Degree of equivalence between INMETRO and KCRV

	-10 ºC	1 ºC	20 ºC
$D_{INMETRO/KCRV}$ / ºC	0.044	0.039	0.012
$U_{INMETRO/KCRV}$ / ºC	0.20	0.21	0.21

The values of $D_{INMETRO/KCRV}$ are all within the k = 2 uncertainty values $U(D_{INMETRO/KCRV})$.

7.6. **INTI/KCRV**

Because INMETRO is linked to the CCT-K6 key comparison reference values at $T_{DP/FP}$ of -10 ºC, 1 ºC and 20 ºC by means of SIM.T-K6.3, the results of INTI in this bilateral comparison (SIM.T-K6.7) for the dew-point temperatures 1 ºC and 20 ºC can be linked to KCRV.

The difference for the dew-point temperatures between INTI and KCRV, $D_{INTI/KCRV}$, is defined as:

$$D_{INTI/KCRV} = D_{INTI\,/\,INMETRO} + D_{INMETRO/NIST} + D_{NIST/KCRV} \qquad (11)$$

Where $D_{INTI/INMETRO}$, the difference for the dew/frost-point temperatures (expressed in ºC) between INTI and INMETRO, is defined as:

$$D_{INTI/INMETRO} = (R_{DP/FP})_{INTI} - (R_{DP\,/\,FP})_{INMETRO} \qquad (12)$$

Where, $(R_{DP/FP})_{INTI}$ and $(R_{DP/FP})_{INMETRO}$ are the measured dew/frost-point temperatures of INTI and INMETRO, respectively.

[7] P. H. Huang, C. W. Meyer, J. D. Brionizio. *Appendix to the Report: Bilateral Key Comparison SIM.T-K6.3 on Humidity Standards in the Dew/Frost-point Temperature Range from –30 °C to 20 °C.* (2017)

The expanded uncertainty of the difference, $U(D_{INTI/KCRV})$, with coverage probability of approximately 95% (k = 2) is defined as:

$$U^2(D_{INTI/KCRV}) = U^2(D_{INTI/INMETRO}) + U^2(D_{INMETRO/NIST}) + U^2(D_{NIST/KCRV}) \qquad (13)$$

Where $U(D_{INTI/INMETRO})$, the expanded uncertainty of the difference with coverage probability of approximately 95% (k = 2), is defined as:

$$U(D_{INTI/INMETRO}) = 2u(D_{INTI/INMETRO}) \qquad (14)$$

The combined uncertainty of the difference, $u(D_{INTI/INMETRO})$, is defined as:

$$u^2(D_{INTI/INMETRO}) = u^2(R_{DP/FP})_{INTI} + u^2(R_{DP/FP})_{INMETRO} + u^2{}_{DRIFT} \qquad (15)$$

Where, $u(R_{DP/FP})_{INTI}$ and $u(R_{DP/FP})_{INMETRO}$ are the uncertainties of the measured dew/frost-point temperatures of INTI and INMETRO, respectively, and u_{DRIFT} is the uncertainty in the comparison due to the drift of the transfer standard.

APPENDIX 1

DETAILS OF PARTICIPATING INSTITUTES

Instituto Nacional de Metrologia, Qualidade e Tecnologia – INMETRO

Address: Laboratório de Higrometria (Prédio 04) – Av. Nossa Senhora das Graças, 50
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Contact: Júlio D. Brionizio
Phone: +55 21 2679 9066
Fax: +55 21 2679 9027
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Instituto Nacional de Tecnología Industrial – INTI

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APPENDIX 2

PROVISIONAL CHECKLIST FOR REPORTING OF CONDITIONS OF MEASUREMENT

The following is guidance for reporting of the background information to the key comparison measurements. This information is likely to be of secondary importance, but will become relevant if there should be any need to resolve anomalies which might appear in the results.

The report should include the following information:

- A full description of the humidity generator used in the comparison and the traceability of the realisation to the SI, including:
    - The gas used (air);
    - The connection between the hygrometer and the standard - tubing material and dimensions;
    - Description of cleaning the mirror;
    - Value of flow rate set for each hygrometer;
    - Description of any problems with the hygrometers, or with the participant's generator system.

- For each separate repetition of each measurement point:
    - Applied reference value(s) (generated dew/frost-point temperature determined by the generator/system, after any correction for pressure drop to the point of use);
    - Standard deviation of the applied value(s);
    - Standard uncertainty of the applied value(s);
    - Values indicated by the travelling standard hygrometer;
    - Standard deviation of the hygrometer indicated values;
    - Difference between the applied (reference system) value and the measured (hygrometer) values;
    - Combined standard uncertainty of the difference;
    - Date when the measurements were carried out;
    - Hygrometer coolant temperature settings and measure values;
    - Temperature and pressure in saturator of generator;
    - Pressure difference between the hygrometer and the generator, and value of correction(s) applied to compensate for this, if any;
    - Environmental conditions (temperature, humidity, pressure);
    - Number of recorded values;
    - Stabilisation time;
    - Time interval taken to record the values;
    - "Raw data" in units of temperature.