COMPARISON OF HUMIDITY STANDARDS

Instituto Nacional de Metrologia, Qualidade e Tecnologia, Brazil
Instituto Nacional de Tecnología Industrial, Argentina
Instituto Nacional de Calidad, Perú

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

TECHNICAL PROTOCOL
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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 (WG 6).

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

1.4. The three 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\(^3\) (INMETRO and INTI) and the first time for INACAL among the participating national metrology institutes, and expand it to a wider range (from -30 °C to +60 °C).

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 Metrología, Qualidade e Tecnologia (INMETRO) – Brazil
- Instituto Nacional de Tecnología Industrial (INTI) – Argentina
- Instituto Nacional de Calidad (INACAL) – Perú.

2.1.2. INMETRO is the Pilot of the comparison, taking main responsibility for running comparison. This comparison will link to CCT-K6 through SIM.T-K6.3.

Because NIST participated in the CCT-K6 multilateral key comparison, some of the results of this bilateral comparison will be linked to the key comparison reference value

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\(^1\) MRA, Mutual Recognition Arrangement, BIPM, 1999.
\(^2\) CIPM MRA-D-05 Version 1.6 March 2016.
(KCRV) for $T_{\text{DP/FP}}$. The degree of equivalence between $T_{\text{DP/FP}}$ realized by a NMI and the KCRV, $D_{\text{NMI/KCRV}}$, is defined as

$$D_{\text{NMI/KCRV}}(T_{\text{DP/FP}}) = [\Delta T_{\text{DP/FP}}]_{\text{NMI}} - [\Delta T_{\text{DP/FP}}]_{\text{KCRV}}$$

Where: $[\Delta T_{\text{DP/FP}}]_{\text{NMI}} = T_{\text{DP/FP}} \text{ (measure or generated by NMI)} - T_{\text{DP/FP}} \text{ (measure by transfer instrument)}$

Because INMETRO participated in the SIM.T-K6.3 some of the results of this bilateral comparison will be linked to the bilateral key comparison reference value (KCRV) for $T_{\text{DP/FP}}$. The degree of equivalence between $T_{\text{DP/FP}}$ realized by INMETRO and NIST, $D_{\text{INMETRO/NIST}}$, is defined as

$$D_{\text{INMETRO/NIST}}(T_{\text{DP/FP}}) = [\Delta T_{\text{DP/FP}}]_{\text{INMETRO}} - [\Delta T_{\text{DP/FP}}]_{\text{NIST}}$$

Where: $[\Delta T_{\text{DP/FP}}]_{\text{NMI}} = T_{\text{DP/FP}} \text{ (measure or generated by NMI)} - T_{\text{DP/FP}} \text{ (measure by transfer instrument)}$

The degree of equivalence between $T_{\text{DP/FP}}$ realized by INTI - INMETRO and INACAL - INMETRO in this comparison, $D_{\text{INTI/INMETRO}}$ and $D_{\text{INACAL/INMETRO}}$, its will be defined as

$$D_{\text{INTI/INMETRO}}(T_{\text{DP/FP}}) = [\Delta T_{\text{DP/FP}}]_{\text{INTI}} - [\Delta T_{\text{DP/FP}}]_{\text{INMETRO}}$$

$$D_{\text{INACAL/INMETRO}}(T_{\text{DP/FP}}) = [\Delta T_{\text{DP/FP}}]_{\text{INACAL}} - [\Delta T_{\text{DP/FP}}]_{\text{INMETRO}}$$

Where: $[\Delta T_{\text{DP/FP}}]_{\text{NMI}} = T_{\text{DP/FP}} \text{ (measure or generated by NMI)} - T_{\text{DP/FP}} \text{ (measure by transfer instrument)}$

Since INTI and INACAL did not participate in CCT-K6 and SIM.T-K6.3, The degree of equivalence between $T_{\text{DP/FP}}$ realized by INTI, INACAL and the KCRV $D_{\text{INTI/KCRV}}$:

$$D_{\text{INTI/KCRV}}(T_{\text{DP/FP}}) = D_{\text{INTI/INMETRO}}(T_{\text{DP/FP}}) + D_{\text{INMETRO/NIST}}(T_{\text{DP/FP}}) + D_{\text{NMI/KCRV}}(T_{\text{DP/FP}})$$

$$D_{\text{INACAL/KCRV}}(T_{\text{DP/FP}}) = D_{\text{INACAL/INMETRO}}(T_{\text{DP/FP}}) + D_{\text{INMETRO/NIST}}(T_{\text{DP/FP}}) + D_{\text{NMI/KCRV}}(T_{\text{DP/FP}})$$

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.

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2.2. **Method of Comparison**

2.2.1. The comparison will be made by 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.

2.3. **Handling of Artefact**

2.3.1. The artefact should 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 should check the contents with the packing list including the operator's manual.

2.3.2. The transfer standard should 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 should be notified immediately before proceeding.

2.4. **Transport of Artefact**

2.4.1. Measurements will start at INMETRO. The travel standard will be hand-carried to INTI and same way to INACAL, where comparison measurements will be conducted at the dew/frost-point temperatures required. The participants should take actions in order to guarantee the exit and entrance of the transfer standard in each participating country.

2.5. **Shipping Costs**

2.5.1. INTI and INACAL are responsible for the cost of shipping including any customs charges and insurance. The insurance should be sufficient to cover the costs of the travelling standard and any damages that could occur.
Timetable

<table>
<thead>
<tr>
<th>Activity</th>
<th>Start Month</th>
<th>Provisional date</th>
</tr>
</thead>
<tbody>
<tr>
<td>Submission of a technical protocol to participants for unanimous approval</td>
<td>May 2016</td>
<td></td>
</tr>
<tr>
<td>Submission of revised technical protocol to SIM/WG3 (thermometry WG) for approval.</td>
<td></td>
<td>December 2016</td>
</tr>
<tr>
<td>Completion of measurements at INMETRO</td>
<td>March 2017</td>
<td></td>
</tr>
<tr>
<td>Travelling standard hand-carried to INTI</td>
<td>March 2017</td>
<td></td>
</tr>
<tr>
<td>Completion of measurements at INTI</td>
<td>April 2017</td>
<td></td>
</tr>
<tr>
<td>Measurements at INMETRO to check the transfer standard stability (if necessary)</td>
<td>May 2017</td>
<td></td>
</tr>
<tr>
<td>Travelling standard hand-carried to INACAL</td>
<td>June 2017</td>
<td></td>
</tr>
<tr>
<td>Measurements at INMETRO to check the transfer standard stability (if necessary)</td>
<td>July 2017</td>
<td></td>
</tr>
<tr>
<td>Report A ready</td>
<td>August 2017</td>
<td></td>
</tr>
<tr>
<td>Deadline for comments on report A</td>
<td>October 2017</td>
<td></td>
</tr>
<tr>
<td>Draft B ready and submitted to SIM/WG3</td>
<td>December 2017</td>
<td></td>
</tr>
<tr>
<td>Paper publication</td>
<td>January 2018</td>
<td></td>
</tr>
</tbody>
</table>

3. DESCRIPTION OF THE TRANSFER STANDARD

3.1. Artefact

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

3.1.2. Details of travelling standard:

<table>
<thead>
<tr>
<th>Manufacturer:</th>
<th>Michell Instruments Ltd., UK</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model:</td>
<td>Optidew Vision</td>
</tr>
<tr>
<td>Serial Number:</td>
<td>118931 (indicator) 118849 (sensor)</td>
</tr>
<tr>
<td>Size in packing case</td>
<td>---</td>
</tr>
<tr>
<td>Owner:</td>
<td>INMETRO, Brazil</td>
</tr>
<tr>
<td>Electrical supply:</td>
<td>90-264 V / 47-440 Hz</td>
</tr>
<tr>
<td>Approximate value for insurance and customs declaration:</td>
<td>US$ 25,000</td>
</tr>
</tbody>
</table>
4. MEASUREMENT INSTRUCTIONS

4.1. Measurement Process

4.1.1. The participants should 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 participant should be informed in order to revise the time schedule, if necessary, as early as possible.

4.1.2. Sample gas generated by a participant's standard/working generator is 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. The sensor of the travelling standard can also be placed directly in the chamber of the generating system without its housing. For dew points near ambient temperature (e.g. +20 °C) normal precautions (heating of pipework) should be used to protect against condensation in sample lines.

4.1.3. A total of six dew-point temperatures humidity levels are used for the comparison at nominal values of 1 °C, 20 °C, 40 °C and 60 °C and frost-point temperature at nominal value 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.

4.1.4. 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. At –20 °C, all data will be assumed to be with respect to ice unless otherwise reported.

4.1.5. Measurements should be made in rising order of dew/frost point.

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

4.1.7. The values of dew/frost point applied to the travelling standard should 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.
4.1.8. 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) Set the indicated flow rate of sample gas from 0.5 to 1 litre per minute;
(3) The dew/frost-point indication of the hygrometer is measured from the hygrometer display or software.

4.1.9. Each dew/frost-point temperature should 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 should be acquired.

4.1.10. 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.

4.1.11. The Pilot will make an assessment of any drift in the travelling standards during the comparison, based on measurements at the Pilot laboratory at the beginning, and end of the comparison period.

4.1.12. 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 should 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 should 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 should 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 should 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-points.

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

5.7. Participants should 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 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. Evaluations should 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 ISO Guide.

6.3. The uncertainty budget stated by the participating laboratory should be referenced to an internal report and/or a published article.
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
Contact: Júlio D. Brionizio
Phone: +55 21 2679 9066
Fax: +55 21 2679 9027
E-mail: jdbrionizio@inmetro.gov.br

Instituto Nacional de Tecnología Industrial – INTI

Address: Física y Metrología – Avenida General Paz, 5445
B1650WAB San Martín – Buenos Aires – Argentina
Contact: Javier García Skabar
Phone: +54 11 4724 6200/300/400
Fax: +54 11 4713 4140
E-mail: jskabar@inti.gob.ar

Instituto Nacional de Calidad – INACAL

Address: Laboratorio de Higrometria – Calle De la Prosa, 150
Lima 41 – San Borja – Lima – Perú
Contact: Billy Quispe Cusipuma
Phone: +51 1 640 8820 extension 1506
E-mail: bquispe@inacal.gob.pe
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.