Consultative Committee for Thermometry (CCT)

Bilateral Comparison of Humidity Standards
National Institute of Standards and Technology, USA
National Metrology Institute of Japan, Japan

3

Frost-Point Temperature –70 °C to -30 °C

Technical protocol

Contents

1. INTRODUCTION

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).

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. A technical protocol had been drawn up by the members of Working Group on Humidity Measurements (WG 6).

The CCT key comparison on humidity standards (CCT-K6) was carried out between 2003 and 2010. The comparison involved 10 National Metrology Institute (NMI) participants, including the National Institute of Standards and Technology (NIST, USA) and the National Metrology Institute of Japan (NMIJ/AIST, Japan). The dew/frost-point values compared in CCT-K6 were –50 °C, –30 °C, –10 °C, 1 °C, and 20 °C. All NMIs participated by comparing their national standard with two common transfer standards. These transfer standards were sent to each NMI at a different time during the course of the key comparison.

NIST performed its CCT-K6 comparisons with the transfer standard between November, 2006 and January, 2007. It used two humidity generators to represent NIST humidity standards. The NIST low frost-point generator was used for the frost points –50 °C and –30 °C, and the NIST hybrid humidity generator (HHG) was used for the dew/frost points –10 °C, 1 °C, and 20 °C. After 2008, however, the bottom limit of the HHG was lowered from –15 °C to –70 °C and the LFPG broke down, requiring a major design change which has not yet been accomplished. Since that time, the HHG has been used to perform all NIST hygrometer calibrations over the range –70 °C to 85 °C. In the spirit of the MRA, NIST proposes to validate the calibration and measurement capabilities (CMCs) of the HHG over the frost-point range –70 °C to –15 °C by performing a bilateral comparison with NMIJ/AIST, which has approved CMCs over this range. As one of the CCT K6 participants NMIJ/AIST can provide linkage of the bilateral comparison results to the CCT K6 key comparison reference values (KCRVs).

This technical protocol has been prepared by the two participants indicated above. 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 practices for the use of dew/frost-point hygrometers.

[1] **MRA, Mutual Recognition Arrangement, BIPM, 1999.**

[2] **T.J. Quinn, "Guidelines for key comparisons carried out by Consultative Committees," Appendix F to the MRA, BIPM, Paris.**

This comparison is aimed at establishing the degree of equivalence between realisations of local scales of dew/frost-point temperature of humid air, in the range –70 °C to –30 °C, among the participating national measurement institutes.

2. ORGANIZATION

2.1 Participants

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

National Institute for Standards and Technology (NIST), pilot	USA
National Metrology Institute of Japan (NMIJ/AIST)	Japan

By their declared intention to participate in this bilateral 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 Bilateral 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 generators/systems.

2.2 Method of comparison

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/working generator using the same measuring process.

Measurements will start at NIST. Comparison measurements will be conducted at the dew/frost-point temperatures required. The transfer standard will then be shipped to NMIJ\AIST, which will conduct similar comparison measurements. Afterwards the transfer standards will be shipped back to NIST, which will perform a second set of comparison measurements to check for drift in the transfer standard. Each participant has 6 weeks to complete measurements.

In case of serious difficulty with customs, or other delays, which might over-run the time period of the ATA Carnet or temporary import licence, the participants may change the schedule of completion.

2.3 Handling of artefact

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 shipping to the next participant. During packing and unpacking, the participants should check the contents with the packing list including the operator's manual.

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

During operation of the transfer standards, 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

The transportation process begins when the artefact leaves the sending laboratory and does not end until it reaches the destination laboratory. All participants should follow the following general guidelines:

(1) Plan the shipment well in advance. The recipient should be aware of any customs issues in their country that would delay the testing schedule. The shipping laboratory must be aware of any national regulations covering the travelling standard to be exported;

(2) Mark the shipping container "FRAGILE SCIENTIFIC INSTRUMENTS" "TO BE OPENED ONLY BY LABORATORY STAFF" and with arrows showing "THIS WAY UP"; attach tip and shock indicators if such devices are available;

(3) Determine the best way to ship the transfer standard to the participant;

(4) Obtain the recipient's exact shipping address. If possible, have it shipped directly to the laboratory;

(5) Coordinate the shipping schedule with the recipient. The sending laboratory should provide the recipient with the carrier, the exact travel mode, and the estimated time of arrival;

(6) Instruct the recipient to confirm receipt and condition upon arrival to the sender. A form for reporting on the receipt of the transfer standard is shown in Appendix 2.

The transfer standard is supplied with its shipping container, which is sufficiently robust to ensure safe transportation.

The artefact will be accompanied by a suitable customs ATA Carnet or temporary import bond (TIB) and documentation uniquely identifying the item. Care should be taken with the timing of the ATA Carnet, which only lasts for one year.

2.5. Shipping Costs

Each laboratory is responsible for the cost of shipping to the other participant, including any customs charges and insurance. The insurance should be sufficient to cover the costs of the transfer standard and any damages that could occur.

2.6. Timetable

Activity	**Provisional date**
Submission of a revised technical protocol to Participants for unanimous approval	December 2014
Submission of revised protocol to CCT for approval	January 2015
Transfer standard sent to NIST for measurements	February 2015
Completion of measurements according to the protocol at NIST	March 2015
Transfer standard sent to NMIJ for measurements	March 2015
Completion of measurements at NMIJ	May 2015
Transfer standard sent back to NIST	June 2015
Completion of Repeat Measurements at NIST	June 2015
Draft A ready	July 2015
Deadline for comments on draft A	August 2015
Draft B ready and submitted to CCT	September 2015

3. DESCRIPTION OF THE TRANSFER STANDARD

The transfer 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.

Details of transfer standard:

Model:	RH 373LX
Serial Number:	xx-xxxx
Size (in Packing case):	63 cm x 53 cm x 40 cm
Weight (in Packing case):	31.2 kg
Manufacturer:	RH Systems, USA
Owner:	RH Systems, USA
Electrical supply:	220 V / 50 Hz
Approximate value for insurance and customs declaration:	US$ xx,xxx

Figure 1. Transfer standard – RH 373LX

4. MEASUREMENT INSTRUCTIONS

4.1. Measurement process

The participants should refer to the operating manual for instructions and precautions for using the transfer 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.

Sample gas generated by a participant's standard/working generator, is introduced into the inlet of a transfer standard hygrometer through a stainless steel tube, terminating with a ¼" VCR fitting for the RH 373LX.

A total of three frost-point temperatures humidity levels are used for the comparison at nominal values of –70 °C, –50 °C, and –30 °C. At –30 °C, all data will be assumed to involve ice condensate on the hygrometer mirror unless otherwise reported. Measurements should be made in the order of rising frost point. The condensate should be cleared and re-formed for each measurement of the frost point.

The values of the frost point applied to the transfer standard should be within ±0.5 °C of the three 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.

The conditions for operation of the transfer standard RH 373LX:

(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 at approximately 0.5 litres per minute;
(3) The frost-point indication of the hygrometer is measured from the hygrometer PRT resistance.

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

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.

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

4.2. Data collection

In the transfer standard, a 100-ohm platinum resistance thermometer (PRT) is embedded beneath the surface of the chilled-mirror to measure the frost-point temperature. The current input to the PRT should be nominally 1 mA. The resistance of the PRT should be measured using a calibrated multi-meter or a resistance bridge, and then converted to a corresponding nominal frost-point temperature using the reference function of IEC 60751 as shown in Appendix 3. This reference function should be used to convert resistance to (arbitrary nominal) temperature.

At each measured value, the mean and standard deviation of multiple readings of the resistance of the PRT should be monitored. Participants may apply their own criteria of stability for acceptance of measurements. When the hygrometer is in equilibrium with the gas sample, the standard deviation of a set of 10 resistance readings, taken over a period of 10 to 20 minutes, is likely to be no more than 0.010 ohms or 0.025 °C approximately.

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

temperature reported for the transfer standard are "arbitrary" values calculated from the measured resistance output. The transfer standard is used simply as a comparator.

5.4 Participants should report results to each other in terms of frost-point temperature. The main measurement results comprise:

- values of frost-point applied to the transfer standard, and associated standard uncertainty
- values of the difference between applied frost point and measured frost point.

A provisional template for reporting results is shown in Appendix 4 and can 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.

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 frost-point apparatus.

6. UNCERTAINTY OF MEASUREMENT

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

- the quoted uncertainty of the frost-point realisation (applied frost point) including any uncertainties due to pressure drop or other influences acting between the point of realisation and the point of use (transfer standard);
- the estimated uncertainty relating to the short-term stability of the transfer standard at the time of measurement;
- the estimated uncertainty due to any drift of a transfer standard over the period of the comparison;
- the estimated uncertainty in mean values due to dispersion of repeated results (reflecting the combined reproducibility of generator and transfer standard);
- the estimated uncertainty due to the resolution of the transfer standard (if found to be significant);
- the estimated uncertainty due to non-linearity of the transfer standard in any case where measurements are significantly away from the agreed nominal value;
- the estimated covariance between applied (generator/system) and measured (transfer 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 Participants are required to submit detailed analyses of uncertainty for their frost-point standards. 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. A provisional template for

documentation of uncertainties is shown in Appendix 6, and can be made available to participants in electronic form as an Excel spreadsheet. Individual institutes may add to the template any additional uncertainties they consider relevant.

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

National Institute for Standards and technology
(NIST) USA

Address: Sensor Science Division (685), 100 Bureau Drive, Stop 8363 Gaithersburg, MD 20899-8363
Contact: Christopher Meyer
Phone: +1 301 975-4825
Fax: +1 301 548 0206
E-mail: cmeyer@nist.gov

National Metrology Institute of Japan
(NMIJ/AIST)
Japan

Address: AIST Tsukuba Central 3, Tsukuba 305-8563, Japan
Contact: Hisashi Abe
Phone: +81-29-861-6845
Fax: +81-29-861-4068
E-mail: abe.h@aist.go.jp

APPENDIX 2. TRANSFER STANDARD RECEIPT FORM

TO: NMIJ (Participating Laboratory)

Fax: +

From: NIST (Participating Laboratory)

Fax: +

We confirm having received the transfer standard of the CCT Bilateral Comparison of Frost-Point Temperatures on.....(date)

After visual inspection

☐ No damage has been noticed;

☐ The following damage must be reported:

.....

.....

.....

Have the hygrometer transportation packages been opened during transit?
e.g., Customs ... Y/N

If Yes please give details.....

Is there any damage to the transportation packages? ... Y/N
If yes please give details.....

Are there any visible signs of damage to the instruments? ...Y/N

If yes please give details.....

Do you believe the transfer standard is functioning correctly? ... Y/N

If not please indicate your concerns .....

Packing list

Received ✓	**Items**	**Dispatched** ✓
	RH 373LX serial number xx-xxxx	
	Manual	
	1 main cable	
	1 resistance measurement cable	
	Customs document on outside of packing case	

Laboratory:.....

Date:..... Signature:.....

APPENDIX 3. IEC 60751 RELATIONSHIP

Based on the IEC 60751 (1995-07), a nominal resistance-temperature characteristic of the PRT in the transfer standard can be defined as follows:

For temperature above 0 °C:

$$R_t = R_0(1 + At + Bt^2) \quad (1)$$

for temperature below 0 °C:

$$R_t = R_0[1 + At + Bt^2 + C(t\text{-}100)t^3] \quad (2)$$

where:

t = temperature (ITS-90), °C,
R_t = resistance at temperature t,
R_0 = nominal resistance of 100 Ω at 0 °C
$A = 3.9083 \times 10^{-3}$ °C^{-1},
$B = -5.775 \times 10^{-7}$ °C^{-2}, and
$C = -4.183 \times 10^{-12}$ °C^{-4}.

APPENDIX 4. TEMPLATE FOR REPORTING OF RESULTS

Refer to Sheet (Measurement results and uncertainties) in accompanying MS Excel file "NIST-NMIJ Comparison.xlsx".

APPENDIX 5. MEASUREMENT CONDITIONS CHECKLIST

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. Reporting of the main results is outlined in Appendix 4.

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;
    - Frequency of AC (or DC) resistance measurement of hygrometer PRTs, and current used;
    - 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 frost-point temperature determined by the generator, after any correction for pressure drop to the point of use);
    - Standard deviation of the applied value(s);
    - Values indicated by the transfer standard hygrometer;
    - Standard deviation of the hygrometer indicated values;
    - Difference between the applied (generator) value and the measured (hygrometer) values;
    - Combined standard uncertainty of the difference;
    - Date when the measurements were carried out;
    - Hygrometer coolant temperature settings;
    - Measured temperatures of RH 373LX pre-cooler temperature;
    - 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 resistance for the PRT measurements.

APPENDIX 6. TEMPLATE FOR DOCUMENTATION OF UNCERTAINTIES

Refer to Sheet (Measurement results and uncertainties) in accompanying MS Excel file "NIST-NMIJ Comparison.xlsx".