APMP Key Comparison of Triple-Point-of-Water Cells APMP.T-K7.2021.1

Technical Protocol

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1. Introduction

After the first CIPM key comparison (KC) of triple-point-of-water (TPW) cells, CCT-K7, was carried out in 2002–2004 [1], an APMP regional comparison was conducted in 2007–2009 with 11 participants. Five of these were participants of the CCT-K7 comparison and, thus, provided the linkage from APMP.T-K7 to CCT-K7. The report of the APMP comparison was published in 2016 [2], and a subsequent bilateral comparison was published in 2018 [3].

A second CIPM KC of TPW cells, CCT-K7.2021, was carried out in 2021–2022 and was published in 2023 [4]. Among the APMP national metrology institutes (NMIs), five laboratories (KRISS, MSL, NIM, NMIA, NMIJ/AIST) participated in this comparison, and their corresponding differences from the key comparison reference value (KCRV) of CCT-K7.2021 ranged from –21.0 μK to +22.4 μK. This is a narrow subrange centred on 0 μK, whereas the total range of differences of all participants ranged from –64.1 μK to +47.0 μK. Thus, any of the five participants from APMP is in a good position to provide a link from the CCT-K7.2021 KCRV to the APMP NMIs.

Discussions on an APMP comparison linking to CCT-K7.2021 started at the 2021 TCT meeting. It was agreed that this comparison should be split into two subgroups of similar sizes (the first designated APMP.T-K7.2021 and the second APMP.T-K7.2021.1). The main reason behind this structure is to reasonably share the workload of the pilot laboratories, where all of the cells are collected and are compared against the pilot's transfer cell. To make use of all five laboratories that participated in CCT-K7.2021, each of the two subgroups would contain two linking laboratories, leaving one NMI available for a future smaller, perhaps bilateral, comparison. It was planned that the second subgroup start the measurements 1–2 years later than the first subgroup, giving opportunity to properly allocate resources for the participants who were not ready to participate right away.

This protocol is based on the APMP.T-K7.2021 protocol, which was made to be as similar as possible to the protocol of CCT-K7.2021. The obvious difference from the CCT-K7.2021 protocol is that the aim of the APMP comparisons is to link to the already-established KCRV of CCT-K7.2021, rather than to calculate new reference values. Another difference is that in the APMP comparisons, we do not seek to explore the link between the previous RMO comparison APMP.T-K7 and these comparisons. Therefore, we are not concerned with whether the participants still have the cells used during APMP.T-K7 (or CCT-K7), but are only interested in the current national standards, transfer cells, and their differences from the CCT-K7.2021 KCRV, calculated via the link laboratories.

MSL is the pilot of this comparison, and both MSL and NMIJ/AIST will provide the link to the KCRV of CCT-K7.2021. The participants in this comparison are CMS, NMC, NMIM, NMISA, NPLI, SCL, and VMI.

2. Objective

The objective is to compare participants' national realisations of the TPW temperature with the CCT-K7.2021 KCRV and determine degrees of equivalence, which can support CMC claims.

3. Participants and Roles

The participant laboratories are listed in Table 1, and the corresponding contact people and their email addresses are listed in Table 2.

Table 1: List of participating institutes, abbreviations used, and their economies.

Institute	**Abbreviation**	**Economy**
Measurement Standards Laboratory of New Zealand	MSL	New Zealand
National Metrology Institute of Japan / National Institute of Advanced Industrial Science and Technology	NMIJ/AIST	Japan
Center for Measurement Standards	CMS	Chinese Taipei
National Metrology Centre	NMC	Singapore
National Metrology Institute of Malaysia	NMIM	Malaysia
National Metrology Institute of South Africa	NMISA	South Africa
National Physical Laboratory, India	NPLI	India
Standards and Calibration Laboratory	SCL	Hong Kong, China
Vietnam Metrology Institute	VMI	Vietnam

Table 2: List of participant laboratories, corresponding contact persons, and their email addresses.

Institute	**Contact Person**	**Contact Email Address**
MSL (pilot and link)	Peter Saunders Ellie Molloy	peter.saunders@measurement.govt.nz ellie.molloy@measurement.govt.nz
NMIJ/AIST (link)	Ikuhiko Saito Januarius Widiatmo Hideki Ogura	saitou.19hiko@aist.go.jp janu-widiatmo@aist.go.jp h.ogura@aist.go.jp
CMS	Shu-Fei Tsai	shu-fei_tsai@itri.org.tw
NMC	Chong Yong Kai	yong_kai@a-star.edu.sg
NMIM	Nurulaini Binti Md Ali	aini@sirim.my
NMISA	Efrem Ejigu	eejigu@nmisa.org
NPLI	Dilip Shivagan	shivagand.nplindia@csir.res.in
SCL	Julian Cheung	cpcheung@itc.gov.hk
VMI	Nguyen Viet Phuong	phuongnv@vmi.gov.vn

4. Comparison Pattern

The comparison will consist of three phases:

1) Each laboratory selects one of its TPW cells for use as a transfer cell and directly compares it against its TPW national reference. The selected transfer cell and the measurement results are delivered to the pilot laboratory.
2) The pilot laboratory compares all transfer cells (including that of the second link laboratory) against the pilot's transfer cell.
3) Each laboratory retrieves its transfer cell from the pilot laboratory and directly re-compares it against its TPW national reference.

5. Timetable

The timetable of the comparison is as follows:

- **1 May 2026:** Start date – the participants select their transfer cell and compare it against their national reference.
- **1 August 2026:** Deadline for the delivery of transfer cells and measurement results (including uncertainty budget) to the pilot lab.
- **1 March 2027:** Deadline for the completion of the pilot laboratory's measurements of all the cells, after which participants' cells can be retrieved.
- **1 June 2027:** Deadline for the delivery of the return measurement results (including uncertainty budget) to the pilot laboratory.
- **1 September 2027:** Deadline for the preparation of the Draft A report.

6. Transfer Cell

The transfer cell shall be carefully selected by each participant according to the following criteria:

- The transfer cell shall be of the highest quality and shall not significantly differ from the quality of the participant's national reference cell(s).
- The transfer cell should preferably be a fused silica cell. If a borosilicate cell is used as the transfer cell, a cell of recent manufacture is preferable.
- If the quality of the cell is suspect on simple inspection or is known to have any kind of abnormal behaviour, it should not be used as the transfer cell.
- The following tests shall be made on the cell and shall be repeated upon reception of the cell by the pilot laboratory:
  - No floating material shall be visible in the water.
  - For the cells with a McLeod gauge or a sufficient remnant "seal-off" tube to trap an air bubble, the compression test described in [5] shall be performed. Prior to testing for air, the cells shall be held vertically at room temperature overnight.
  - There shall be a sharp "click" audible when the cell is gently inverted, indicating a very low amount of residual air ("water hammer test").

The pilot reserves the right to reject transfer cells that do not meet the minimum selection criteria when tested on receipt. Laboratories normally using other tests are invited to apply them in addition and to describe them. Laboratories are asked to provide information about the dimensions (in cm) of the chosen transfer cell as soon as possible. This particularly applies to cells with unusual dimensions (for example, very large or very small cells).

Participant laboratories are free to select and measure an additional cell to keep as a back-up in case problems (or breakage) arise with their transfer cell.

6.1 Distinction between national reference cells and transfer cell

National reference cell(s): The ensemble of cells, or a single cell, used to maintain 273.16 K in the participant's economy. Accordingly, it should have all practical corrections applied and the uncertainty analysis should include all sources of uncertainty. Each participant is required to submit the uncertainty budget for the difference between their transfer cell (see below) and national reference, as well as a detailed description of the national reference: single cell or ensemble; whether isotope and impurity corrections are applied; fused silica or borosilicate; natural water or spiked; age of cells.

Transfer cell: The single cell sent to the pilot laboratory. Each participant should report the temperature difference between the temperature realised by this cell and their national reference (note, the transfer cell may also be part of the national reference). The temperature realised by the transfer cell should be corrected only for effects associated with self-heating and hydrostatic pressure (other corrections, such as for isotopes and impurities should not be applied to the transfer cell measurements). Each participant is required to submit an uncertainty budget for the measurement of the temperature difference (i.e., between the partially-corrected transfer cell and the fully-corrected national reference).

7. Shipment of the Transfer Cell

The packing of the transfer cell, before the shipment to the pilot laboratory and back to the participating laboratory, falls under the responsibility of the participating laboratory. The participating laboratory shall select and apply the preferred packing method before shipping the transfer cell to the pilot laboratory, and provide detailed information to the pilot for packing the transfer cell before the return shipping.

One method for packing TPW cells is described in MSL Technical Guide 44 "Shipping TPW Cells" [6]; however, such a method is suitable only for TPW cells not having a McLeod gauge.

Some guiding principles in packing TPW cells are reported below:

- Use very large wooden crates, so that they can be only gently handled by forklifts.
- Use a large soft sponge layer to reduce the *g*-forces experienced by the cell during shipping.
- The cell should be placed along the diagonal of the crate to prevent water hammer effects.

The participant laboratory is also responsible for:

- Making proper arrangement for customs formalities (e.g., ATA carnet).
- The transfer costs, customs charges, and any damage that may occur during transport.
- If deemed necessary, taking out insurance for the transport of the transfer cell.

8. Measurement Instructions and Procedure

Each laboratory must carefully select its transfer cell according to the criteria given in Section 6 and compare it against its national reference (single cell or set of cells). The measurements shall be performed on two separately prepared ice mantles of the transfer cell. The participant is free to use their preferred method for the preparation of the ice mantle, but the measurements should not start earlier than 7 days after preparation of the ice mantle. Depending on the local preparation technique, the minimum waiting time required might be longer than 7 days. A minimum of 10 measurements per mantle (one per day) shall be reported in the appropriate Measurement Report Form. Before each measurement, an inner melt shall be induced. The recommended method for inducing the inner melt is insertion of a room-temperature metal or glass rod in the thermometer well for a few seconds. The ice mantle should then freely rotate around the well when a gentle rotational impulse is given to it. Apart from this, the measurement procedure shall be the one normally applied by the laboratory. The distance of the mid-point of the sensor from the top of the ice mantle for each measurement shall be reported and shall be used to calculate the hydrostatic correction.

If a laboratory uses special parts with its transfer cell, like bushing or a foam pad, this should also be sent to the pilot, together with a short description of its use.

For each transfer cell, an immersion profile shall be provided to ensure that the thermometer senses only the temperature of the ice/water interface and is not influenced by ambient conditions. For each step of the profile, the self-heating correction shall be determined and applied. It is recommended that a step height of 1 cm or 2 cm is used, and the measurements are taken up to about 10 cm above the lowest sensor position.

After return of the transfer cell from the pilot laboratory, the full set of measurements described above shall be repeated. If the transfer cell is found to be unstable or is broken during the return travel or measurements, this information shall be immediately given to the pilot. In this case, the pilot will evaluate the stability of the transfer cell during the measurements at the pilot laboratory. If the transfer cell was stable during the measurements at the pilot laboratory, only the measurements performed by the participants before delivering the transfer cell to the pilot will be used in the analysis of the results.

The pilot shall select its own transfer cell and compare it against its national reference. Subsequently, all transfer cells delivered to the pilot shall be compared against the pilot's transfer cell and that of the second link laboratory. For each transfer cell, similarly to the measurements at the participant institute, a minimum of 10 measurements shall be performed on each of two separately prepared ice mantles. The ice mantles shall be prepared with the technique used by the pilot. The waiting time before starting the measurements after the preparation of the ice mantle shall be at least 7 days. Each measurement day, the pilot's transfer cell, NMIJ/AIST's transfer cell (the second link laboratory), and a number of participants'

transfer cells shall be measured. The number of transfer cells measured daily will depend on the TPW storage capability of the pilot laboratory.

9. Reporting the Results

Each laboratory must report the performed measurements by filling in the appropriate parts of the Measurement Report Form, which is an integral part of this protocol. The Measurement Report Form is an Excel file, composed of 8 sheets:

- In the 1st sheet, named "**Participant**", the participant laboratory must insert the information on the participant laboratory and the contact person(s).
- In the 2nd sheet, named "**Equipment**", the participant laboratory must insert information on the equipment used for the measurements.
- In the 3rd sheet, named "**Transfer cell**", the participant laboratory must insert information on the selected (and measured) transfer cell.
- In the 4th sheet, named "**National reference**", the participant laboratory must provide detailed information on the national reference.
- In the 5th sheet, named "**Results 1st mantle**", the participant laboratory must provide detailed information on the results of the measurements performed on the 1st mantle of the transfer cell.
- In the 6th sheet, named "**Results 2nd mantle**", the participant laboratory must provide detailed information on the results of the measurements performed on the 2nd mantle of the transfer cell.
- In the 7th sheet, named "**Immersion profile**", the participant laboratory must provide detailed information on the results of the immersion profile measurements performed on the transfer cell.
- In the 8th sheet, named "**Uncertainty budget**", the participant laboratory must provide a detailed uncertainty budget for the measurement of the difference between the transfer cell and the national reference, including uncertainty components arising from the realisation of the national reference. The major uncertainty components are listed in the Excel sheet. The participant laboratory is free to modify the uncertainty budget in the Excel sheet. The uncertainty budget must satisfy the following requirements:
  - Be compliant with the general rules of the "Guide to the expression of uncertainty in measurement" (GUM) [7].
  - In order to avoid double-counting of uncertainty sources, each uncertainty component listed in the budget must specify its physical cause or causes.
  - Related to the previous point, repeatability and reproducibility, without clarifying exactly which physical causes they originate from, are not acceptable as uncertainty sources.

10. Communication Flows

The participant laboratory must promptly communicate to the pilot:

- The dimensions of the transfer cell, including the inner diameter and length of the re-entrant well.

- Any unexpected delay that prevents the participant laboratory from delivering the transfer cell and/or measurement results by the deadline reported in the comparison timetable.
- The date of shipment of the transfer cell to the pilot's laboratory and the expected date of the arrival of the transfer cell at the pilot's laboratory.
- Detailed instructions to the pilot for packing the transfer cell before the return shipping.

The pilot must promptly communicate to the participant laboratory:

- The reception of the transfer cell and any visible damage to the transfer cell.
- The procedure in the case of unexpected delay at the participant laboratory.

11. Analysis of the Key Comparison Results

MSL and NMIJ/AIST will provide a link between the results of this comparison and the KCRV from the CCT-K7.2021 comparison. The final report of CCT-K7.2021 [4] contains the values of the differences $T_{\mathrm{nr,MSL}} - T_{\mathrm{KCRV}}$ and $T_{\mathrm{nr,NMIJ}} - T_{\mathrm{KCRV}}$, along with their uncertainties, where $T_{\mathrm{nr,MSL}}$ and $T_{\mathrm{nr,NMIJ}}$ are the temperatures of MSL's and NMIJ/AIST's national references, respectively, and T_{KCRV} is the value of the KCRV in the CCT-K7.2021 comparison.

The current comparison aims to determine the degrees of equivalence for each participant NMI, $\mathrm{DoE}_{\mathrm{NMI}} = T_{\mathrm{nr,NMI}} - T_{\mathrm{KCRV}}$ (including its uncertainty), via the links provided to T_{KCRV} from MSL and NMIJ/AIST, where $T_{\mathrm{nr,NMI}}$ is the temperature of the national reference of the participant NMI. For example, the link via MSL can be established as:

$$\left(T_{\mathrm{nr,NMI}} - T_{\mathrm{KCRV}}\right)_{\mathrm{MSL}} = \left(T_{\mathrm{tr,NMI}} - T_{\mathrm{tr,MSL}}\right) + \left(T_{\mathrm{tr,MSL}} - T_{\mathrm{nr,MSL}}\right) - \left(T_{\mathrm{tr,NMI}} - T_{\mathrm{nr,NMI}}\right) + \left(T_{\mathrm{nr,MSL}} - T_{\mathrm{KCRV}}\right), \tag{1}$$

where $T_{\mathrm{tr,NMI}}$ and $T_{\mathrm{tr,MSL}}$ are the temperatures of the transfer cells of the participant NMI and MSL, respectively. Each set of brackets on the right-hand side of this equation corresponds to a temperature difference: the first bracket is the difference between the participant NMI's transfer cell and MSL's transfer cell, as measured by MSL (the pilot laboratory) after receiving the cell from the participant; the second bracket is the difference between MSL's transfer cell and its national reference, as measured by MSL before receiving the cells from the participants; the third bracket is the difference between the participant NMI's transfer cell and its national reference, as measured by the participant NMI—this difference is submitted as a result to the pilot laboratory; the fourth bracket is the difference between MSL's national reference and the KCRV of CCT-K7.2021, as published in the final report [4] of that comparison.

A similar equation can be written for the link via NMIJ/AIST:

$$\begin{aligned}\left(T_{\mathrm{nr,NMI}} - T_{\mathrm{KCRV}}\right)_{\mathrm{NMIJ/AIST}} = {} & \left(T_{\mathrm{tr,NMI}} - T_{\mathrm{tr,NMIJ/AIST}}\right) + \left(T_{\mathrm{tr,NMIJ/AIST}} - T_{\mathrm{nr,NMIJ/AIST}}\right) \\ & - \left(T_{\mathrm{tr,NMI}} - T_{\mathrm{nr,NMI}}\right) + \left(T_{\mathrm{nr,NMIJ/AIST}} - T_{\mathrm{KCRV}}\right).\end{aligned} \tag{2}$$

Here, the first bracket on the right-hand side is measured by MSL, the second is submitted by NMIJ/AIST, the third is submitted by the participant NMI, and the fourth is from the CCT-K7.2021 final report.

The degrees of equivalence, $\mathrm{DoE}_{\mathrm{NMI}}$, will be calculated as the mean of equations (1) and (2):

$$\mathrm{DoE}_{\mathrm{NMI}} = \frac{\left(T_{\mathrm{nr,NMI}} - T_{\mathrm{KCRV}}\right)_{\mathrm{MSL}} + \left(T_{\mathrm{nr,NMI}} - T_{\mathrm{KCRV}}\right)_{\mathrm{NMIJ/AIST}}}{2},$$

and the uncertainties will be propagated according to the GUM [7].

A consistency check for the two link laboratories will also be carried out. That is, it will be checked whether $T_{\mathrm{nr,NMIJ/AIST}} - T_{\mathrm{nr,MSL}}$ determined during the CCT-K7.2021 comparison is consistent with the value of this difference measured in the current comparison. The CCT-K7.2021 difference [4] is simply

$$\begin{aligned} T_{\mathrm{nr,NMIJ/AIST}} - T_{\mathrm{nr,MSL}} &= \left(T_{\mathrm{nr,NMIJ/AIST}} - T_{\mathrm{KCRV}}\right) - \left(T_{\mathrm{nr,MSL}} - T_{\mathrm{KCRV}}\right) \\ &= 22.4\ \mu\mathrm{K} - 14.1\ \mu\mathrm{K} \\ &= 8.3\ \mu\mathrm{K}. \end{aligned}$$

The value from the current comparison will be determined as:

$$T_{\mathrm{nr,NMIJ/AIST}} - T_{\mathrm{nr,MSL}} = \left(T_{\mathrm{tr,MSL}} - T_{\mathrm{nr,MSL}}\right) + \left(T_{\mathrm{tr,NMIJ/AIST}} - T_{\mathrm{tr,MSL}}\right) - \left(T_{\mathrm{tr,NMIJ/AIST}} - T_{\mathrm{nr,NMIJ/AIST}}\right).$$

If the consistency check fails, the pilot will discuss the issue with NMIJ/AIST and decide how to proceed. The likely solution will be to add a value corresponding to the change in the appropriate national reference since the CCT-K7.2021 comparison, $T^{\mathrm{APMP}}_{\mathrm{nr,MSL}} - T^{\mathrm{CCT}}_{\mathrm{nr,MSL}}$ and/or $T^{\mathrm{APMP}}_{\mathrm{nr,NMIJ/AIST}} - T^{\mathrm{CCT}}_{\mathrm{nr,NMIJ/AIST}}$, to equations (1) and/or (2), respectively.

References

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