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Comparison of Five Isotope-Corrected Water-Triple-Point Cells with the NMIA-2002 WTP Ensemble

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Abstract A comparison of NMIA's new water-triple-point (WTP) ensemble with a previously established ensemble is reported. Until 2007, the kelvin in Australia was defined as the average of an ensemble of WTP cells that were selected for stability and purity and collected over a period of several years from a variety of sources. As a result of the recent CCT-K7 comparison, a clarification of the SI definition for the kelvin was adopted, explicitly specifying the isotopic composition of the water in WTP cells. Although NMIA's results were within the estimated uncertainties, NMIA initiated a project to acquire cells with isotope information from several manufacturers and batches to establish a new ensemble. We find that the standard deviation of the isotope-shift-corrected temperatures of five cells from three manufacturers to be 6 μK, which is significantly lower than that of the cells in the previous ensemble, which was 24 μK. The average temperature of the new ensemble is found to be approximately 107 μK higher than that of the previous ensemble. This difference is consistent with the findings of CCT-K7, which identified a group of laboratories controlling isotope effects, and is displaced 73 μK from the mean of the other laboratories.

Keywords Cell ensemble · Isotopic correction · Kelvin definition · Triple point of water

1 Introduction

The SI unit of temperature, the kelvin, is defined as 1/273.16 of the temperature of the triple point of water, where the solid, liquid, and gaseous forms of water exist in thermal equilibrium. This is realized practically in a so-called water-triple-point (WTP)

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cell, a sealed glass ampoule of several hundred milliliters of very high-purity, degassed water. Since 2006, the isotopic composition of the water has also been prescribed as that of "the composition of the International Atomic Energy Agency reference material Vienna Standard Mean Ocean Water (VSMOW)" with the agreed composition: D/H = 0.00015576, $^{18}O/^{16}O = 0.0020052$, and $^{17}O/^{16}O = 0.0003799$.

The actual temperature realized by WTP cells is affected by several factors; the major contributions arise from:

1. the chemical purity of the water sample
2. the isotopic concentration (H, D, O^{16}, O^{18})
3. residual gases in the cell
4. stray thermal fluxes (conduction errors)
5. stresses and defects in the ice crystals
6. the vertical water pressure variation in the cell due to gravity

As the cells are completely sealed once manufactured, it is difficult to confirm the magnitude of any systematic errors arising from the first two of these terms, which usually dominate the uncertainty estimate for the cell. As most laboratories use commercially procured cells, they are generally reliant on statements or claims made by the manufacturers.

In Australia, we try to assess the contribution of these error sources by maintaining an ensemble of cells, from a variety of sources (each of which claims the cells to be a "true" WTP realization), and collected over several years. At regular intervals, we measure the dispersion of the temperatures realized by the cells, and *define* the average of the ensemble to be exactly 273.16 K. Each cell is then assigned a value with respect to this.

Prior to 1996, NMIA (then NML) defined a single cell from the available cells as the Australian realization, with an estimated error based on ad hoc comparisons with other cells held by the laboratory.

The first formal comparison of NMIA WTP cells occurred in 1996 [1]. Twenty WTP cells held at the NMI were compared, four cells at a time, using an ice bath. Of these, one broke during testing and 11 were discarded, based on either the presence of residual gas or significant electrical conductivity, according to an in situ measurement [2]. The remaining set of eight cells was identified as suitable for use in forming an ensemble of cells to define the NMI kelvin. For each cell, measurements were made on a single ice mantle, which was allowed to anneal (at 0 °C) for only 24 h. Consequently, no estimate of the contribution of the reproducibility of the ice mantle to this variance could be established, and the standard deviation of the ensemble, 75 μK, was relatively large compared to later measurements.

In July–September 2000, another comparison of the NMI cells was made [3,4]. Seven cells with a measured standard deviation (SD) of 31 μK were chosen for the ensemble to realize the kelvin. In this study, an extensive investigation of reproducibility and sources of measurement error was made. Two ice mantles were measured on each cell, with three days allowed to anneal the mantle after freezing.

In August 2002, a third comparison [5,6] was made. Eight cells, with a measured standard deviation of 24 μK, were chosen to realize the kelvin. In this comparison, five ice mantles per cell, each allowed 10 days to anneal, were measured. A new

cell-maintenance bath was designed and built, allowing all eight cells to be directly compared (rather than in batches of four as in the 1996 and 2000 realizations).

The present realization, based on measurements in Oct–Dec 2006, uses five cells each with a reported isotopic concentration. A similar procedure to the 2002 measurements, with five mantles, ten-day annealing, and a water bath to simultaneously measure all the cells, was used. Two cells from the old 2002 realization were also measured to provide a link to the previous realization.

2 Maintenance Bath for WTP Cells

After each WTP cell is pre-cooled to 0 °C, an ice "mantle" 5 mm to 10 mm thick is frozen over the thermometer well. The ice mantle must be left to stabilize for several days to sufficiently anneal stresses and defects in the ice, as they decrease the apparent temperature. During this time, the volume of ice surrounding the thermometer well stays constant but the ice crystal domains grow to several mm in size.

A simple WTP water bath fabricated by NMIA (Fig. 1) is able to maintain ice mantles on nine cells for several months without significant melting or freezing. A stainless-steel box internally 72 cm deep with a 40 cm square base, insulated by 10 cm of polystyrene, is filled with water. A few liters of ethanol is added to prevent icing of the cooling coils. A bubbler with 15 $L \cdot min^{-1}$ flow of air is used to stir the water. A commercial water circulator circulating water/alcohol through several meters of copper tubing cools the bath. The out-of-balance signal from a platinum resistance

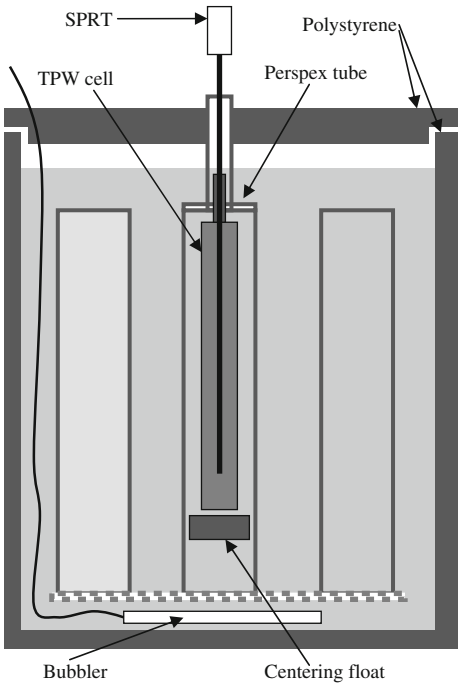

Fig. 1 Schematic diagram of the WTP maintenance bath

thermometer connected to a Leeds and Northrup 8078 resistance bridge is used as the set temperature for the circulator (i.e., 10 mK out-of-balance changes the circulator by 1.6 °C). This simple system achieves a stability of 0.1 mK and a uniformity of 2 mK. The bath is usually set to between 0.0 and 0.01 °C. Two independent "policeman" controllers are set to shut off the cooler if the bath temperature falls below −0.025 °C.

Nine Perspex tubes, with an inner diameter of 50 mm and a length of 490 mm, are mounted onto a frame within the bath. The tops of the tubes are covered by Perspex lids 5 cm below the surface of the water, to allow the thermometer stem to conduct heat to the bath water rather than to the WTP cell. WTP cells are placed within each Perspex tube, and a small polystyrene block under each cell centers it and prevents it from sinking to the bottom of the Perspex tube. The Perspex tubes ensure a layer of stagnant water around the cell, thus reducing the heat transfer to the cell. Measurements indicate that the time constant for thermal equilibration between the cell holder and the water bath is 45 min.

3 Measurement Procedure

The protocol used for comparison of the cells is described below:

- Cells were frozen using a small Freon heat pipe inserted into the thermometer well that was cooled by dry ice using the technique described in [1,3,5,7], forming an ice mantle typically 5 mm to 10 mm thick.
- Cells were left to anneal for 10 days in the water bath.
- A small sponge, 1 mm to 2 mm thick, was placed in the bottom of each triple-point well to prevent the thermometer from directly touching the base of the well (the wells are fully filled with water as the cells are submerged in the bath).
- The height of water above the base of the well in each cell was measured using a glass rod with a graduated scale. The water level above the base of the well could be determined with an uncertainty of approximately 1 mm.
- A thin layer of water was melted around the thermometer well by inserting, in succession, three glass rods at room temperature, allowing 1 min each for equilibration. The thermometer is thus surrounded by two concentric liquid–solid interfaces.
- A quartz-sheathed 25 Ω standard platinum resistance thermometer (SPRT) was inserted into the cell, and allowed 10 min to stabilize.
- 30 ASL F18 bridge readings (75 Hz, 0.1 Hz bandwidth) were taken, 10 at each of three sensing currents (1 mA, $\sqrt{2}$ mA, 1 mA), after allowing 2 min to stabilize at each current, to determine a self-heating-corrected thermometer resistance (self-heating was typically 1.2 mK). Establishing a liquid layer and measuring the SPRT resistance at the three sensing currents took 25 min.
- Over a two-day period, the thermometer was circulated through all the cells in the bath four to six times.
- The cells' ice mantles were melted by warming the cells in lukewarm water, and new mantles were frozen.
- The procedure above was repeated four times to obtain data on five mantle realizations.

Allowing for annealing and measuring time, each mantle assessment took two weeks, so the assessment of the ensemble took 10 weeks in total, and resulted in typically 25 individual self-heating-corrected measurements on each cell.

4 Analysis of Variance Sources

In previous kelvin realizations at NMIA [3,5], an experimental investigation of the sources of measurement noise and possible systematic error was undertaken. For example, no significant difference in the measured self-heating was observed if the time allowed for stabilization after changing bridge currents was significantly increased.

In the measurements here, the measured standard deviations of the three sets of 10 individual resistance bridge measurements is used to calculate the expected variation (SEM) of the self-heating-corrected SPRT resistance value obtained from the 30 bridge values, giving a value of typically 9 μK to 12 μK. Over the two days of measurements on a given mantle, the measured SD of the five sets of measurements on a given mantle is consistent with this. The predicted variability in the mean of these five measurements of a single mantle with respect to the average is thus $1/\sqrt{5}$ of this, or about 4 μK. The variation in temperature differences is thus fully attributable to the measured AC bridge noise measurements alone. However, when the cells are melted and refrozen to give five independent mantles, the measured SD varies from 4 μK to 18 μK, which is larger than could be expected statistically, based purely on the measured electrical noise. The process of melting and re-freezing the WTP mantle clearly results in an additional source of variance for some cells, presumably due to the geometry of the cell and the re-entrant thermometer well.

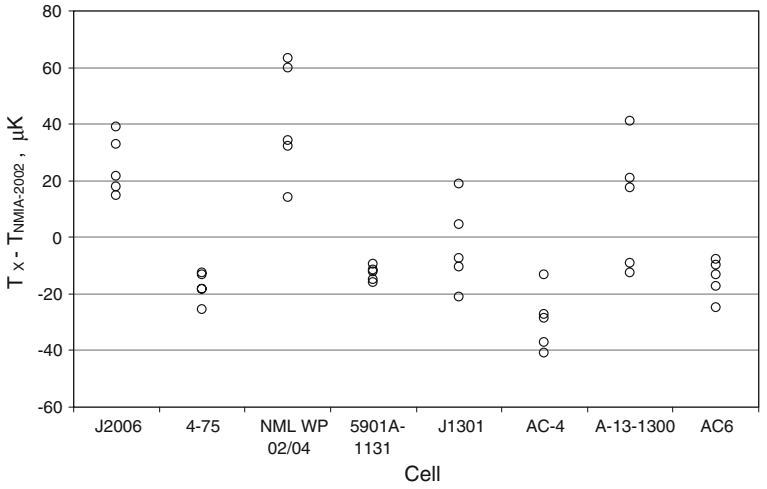

Fig. 2 Comparison of the assigned temperatures of the eight cells used to realize the NMIA-2002 definition of the kelvin for Australia. The electrical noise on each point corresponds to a variance of typically 10 μK

In the NMIA-2002 [5] realization, shown in Fig. 2, a similar finding was made, with some cell variability (SD of re-frozen mantles) ranging from 4 µK to 21 µK. In the NMIA-2000 realization [3], cell variability was typically 21 µK for all cells, which was larger than could be explained by any electrical noise or thermometer instabilities. The improvement between the 2000 and 2002 studies is attributed to the longer annealing period used for the two recent studies.

5 Hydrostatic Tracking

Because water expands on freezing, the temperature of an ice–water interface decreases with increasing hydrostatic pressure (Clausius–Clapeyron relationship from thermodynamics). This results in a 7.3 µK · cm^{-1} decrease in temperature with depth into the water of the cell. Figure 3 shows that the measured temperature profile within the wells of four cells closely tracks the predicted theoretical relationship. However, the deviations (up to 30 µK for individual measurements) from the theoretical slope exhibit slightly more variance than could be explained by the electrical noise (SD of 9 µK to 12 µK) alone. Repeated measurements tracking the hydrostatic depression curve on a given mantle showed these deviations were not reproducible and are attributed to variation in the thermal contact between the well and thermometer (there is typically a 0.5 mm to 1 mm gap between them). As this effect is not fully understood, a 20 µK rectangular half-width uncertainty is adopted as an uncertainty estimate. The systematic error in the temperature differences between cells arising from the shunting effect of moisture within the SPRT is also assessed by deviation from the hydrostatic depression curve; however, in this facility, the curve can be tracked out to 250 mm [5], suggesting any such errors are negligible.

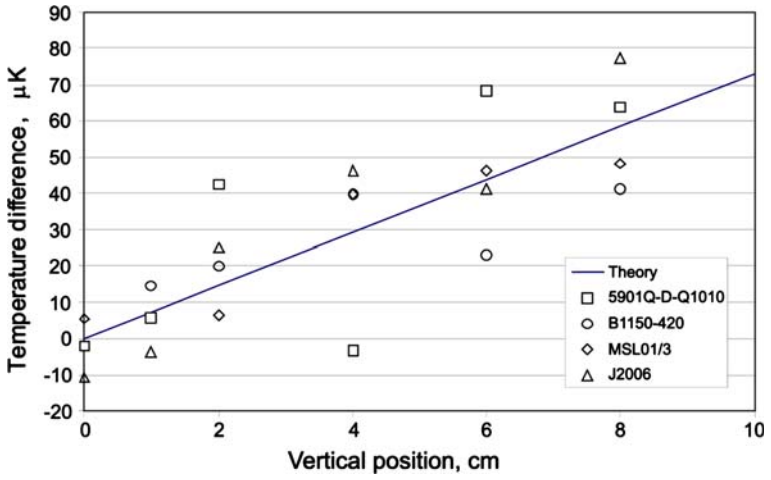

Fig. 3 Measured temperature profile of four of the cells used in this study, together with the theoretical 7.3 µK · cm^{-1} hydrostatic-pressure temperature gradient (*solid line*)

6 Definition of the NMIA Kelvin

The details of the five cells used for the 2006 ensemble are given in Table 1, together with the isotopic composition of the cell as provided by the supplier. We have adopted the coefficients presented in [8] to correct the temperatures of each cell to the new SI definition, and applied corrections for the measured height from the sensor midpoint to the water level in the cell. For each mantle on a cell, we *define* the average of the isotope and hydrostatic-corrected temperatures of the five cells to be exactly 273.16 K. Note that we do not calculate an actual "ensemble" average temperature, only differences of each cell from the average of the cells. We consider each cell in the ensemble to have equal weighting in the ensemble. This has been repeated four times on the set of cells, and Fig. 4 shows the five mantle realizations as five data points for each cell. The mantle reproducibility of each cell is estimated from the standard deviation of these five points, u_A (cell i). Taking now the average of the five mantles on each cell (Table 2) to give the final temperature estimates for each cell, we find the measured standard deviation of these five final temperature estimates to be u_B (ensemble) = 5.7 μK, and this variance may be taken as an experimental estimate of the contribution from the following uncertainty components:

1. Electrical noise
2. Self-heating
3. Stray thermal fluxes
4. Impurities
5. Uncertainty in measurement and correction of isotopic effects
6. Hydrostatic head errors

In the case of the measured isotopic concentrations, the two MSL cells were measured by the New Zealand Institute for Geological and Nuclear Sciences, the Hart Scientific cell measured by the SIRFER facility at the University of Utah, and the values for the two Isotech cells were those reported by Isotech. In the NMIA-2002 realization, we had assumed that the systematic errors attributed to these effects were fully uncorrelated, and adopted the standard error in the mean (SEM). However, as will be seen later, this assumption resulted in an underestimate of the effect of isotopic variation. In the NMIA-2006 ensemble, we allow that there may be some degree of correlation within the measured variance, and adopt the (larger) experimental standard deviation of the five cells. This variance will implicitly include the measurement uncertainty of

Table 1 Source and claimed isotopic deviation from VSMOW for cells used in the NMIA 2006 WTP ensemble

Cell	Source	δ (D/H)	δ ($^{18}O/^{16}O$)	Calculated isotopic correction, μK
5901D-Q1010	Hart Scientific	0.0006	−0.0006	0.0
B11-50-420	Isotech	−0.01064	-8.10×10^{-4}	6.8
MSL01/3	MSL	−0.0906	−0.0147	62.6
MSL04/2	MSL	−0.0562	−0.0095	39.1
B1150Q588	Isotech	0.004060	0.025150	−17.5

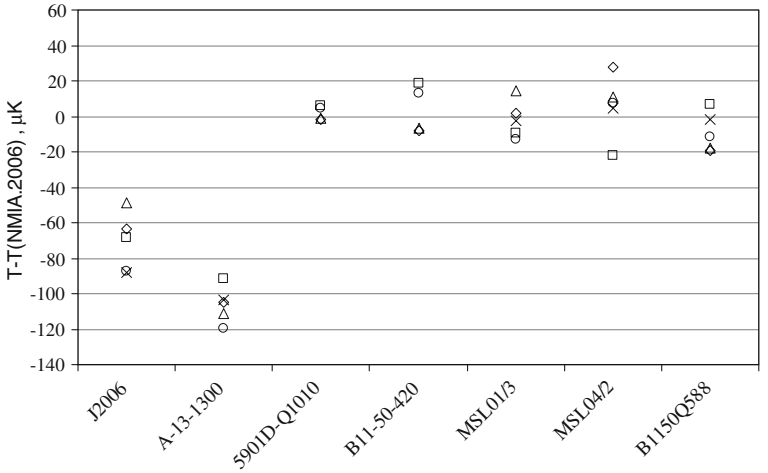

Fig. 4 Measured temperatures of the five cells in the NMIA-2006 ensemble (corrected for isotopic composition and hydrostatic head), and two cells (J2006 and A-13-1300) from the NMIA-2002 ensemble. The electrical noise of each point corresponds to a variance of typically 10 µK

Table 2 Temperatures assigned to each cell (isotope- and hydrostatic head-corrected) in the NMI-2006 WTP ensemble with respect to the NMI-2006 ensemble average

Cell	Assigned average temperature $T_i - T_{\mathrm{NMI2006}}$ (µK)	Standard deviation of five mantles $u_{\mathrm{A}}(T_i)$ (µK)	Total expanded uncertainty in assigned temperature (µK) at 95 % C.L.
J2006[a]	−71.0	16.8	31.1
A-13-1300[a]	−106.0	10.3	28.4
5901D-Q1010	2.0	4.0	27.1
B11-50-420	4.2	13.7	29.6
MSL01/3	−1.8	10.9	28.6
MSL04/2	5.5	17.8	31.6
B1150Q588	−8.8	11.1	28.7

[a] Cells from 2002 ensemble for comparison

the individual isotopic measurements. Note that the estimated uncertainty in isotopic concentration given by the suppliers corresponds to an uncertainty of a few µK. If the correction for the isotopic concentration is not made, the standard deviation of the five cells increases substantially, from 5.7 µK to 30 µK. This latter value is similar to the standard deviations of the NMIA-2000 and NMIA-2002 sets of cells, which suggests that the manufacturing process for the two sets of cells is similar.

For any given cell compared to the ensemble average, the uncertainty in the assigned average cell temperature, T_{k}, includes the following components:

1. The experimental SD of the mean of the temperatures of the five cells in the ensemble: u_{B} (ensemble)
2. The experimental SD of the means of the temperatures obtained from the five mantles on each cell, i.e., experimentally determined mantle reproducibility of the cell: $u_{\mathrm{A}}(\text{cell } i)/\sqrt{5}$

3. An estimated ±10 mm uncertainty in the effective sensing position of the SPRT element (which is 40 mm long): $u_{\text{hydro}} = 7.3\,\mu\text{K}/\sqrt{3}$
4. Stray heat fluxes, estimated from the deviation from the theoretical hydrostatic temperature gradient: $u_{\text{heatflux}} = 20\,\mu\text{K}/\sqrt{3}$

These uncertainties are combined using the "ISO-GUM" approach [9]. The temperature assigned to each cell and its expanded uncertainties (note: $k \approx 2.0$) is given in Table 2, together with the measured reproducibility of its mantle. The raw data for the NMIA-2006 study are available in [10].

7 Comparison with Previous Realizations

Table 3 compares the four NMIA realizations, showing the improvement in the uncertainty of the NMIA definition (61 µK → 29 µK → 20 µK → 15.9 µK), and the assigned temperature of a given cell in the ensemble (80 µK → 44 µK → 30 µK → 30 µK).

There were six common cells between the "2000" and "2002" kelvin ensembles and four cells in common between the "1996" and "2002" ensembles. These common cells allowed us to check the effective drift of the NMIA definition (Table 3). Two cells, J2006 and A-13-1300, from the 2002 ensemble were measured against the five cells of the new 2006 ensemble, allowing determination of the difference between the ensembles. The measured temperature difference between the two link cells was 36 µK, consistent with the 14 µK difference determined in 2002, suggesting that the link cells were stable. The uncertainty in the difference between the 2002 and 2006 ensembles is taken as the quadrature sum of the uncertainties in the assigned temperatures of the link cells in 2002 and 2006. The 2006 ensemble was found to be 97 µK ($U_{95} = 43\,\mu\text{K}$) higher via cell J2006 and 118 µK ($U_{95} = 43\,\mu\text{K}$) higher via cell A-13-1300. These differences are in good agreement. Assuming these two differences to be uncorrelated, the estimated difference between the 2002 and 2006 ensemble realizations is 107 µK ($U_{95} = 31\,\mu\text{K}$).

The differences in temperature between the 1996, 2000, and 2002 ensembles are less than the variances within each ensemble, which supports the hypothesis that the cells from these three ensembles have come from the same population. However, the difference between the 2002 and 2006 ensembles significantly exceeds the variance within both ensembles, indicating that these two sets of cells are from different

Table 3 Comparison of NMIA WTP ensembles

Year:	1996	2000	2002	2006
# of cells n	8	7	8	5
SD of n cells (µK)	75	31	24	5.7
U_{95} (µK)	61	29	20	15.9
Typical U_{95} of a given cell (µK)	80	44	30	30
Apparent difference to 2002 average (µK)	−27 SD 39 µK over four common cells	−6 SD 9 µK over six common cells	Reference	+107 $U(k = 2) = 31\,\mu\text{K}$ (mean of two common cells)

populations, which should not be surprising since the 2006 cells have been corrected for isotopic composition. However, it is worthwhile to note that at the time the 2002 cell ensemble was established, based on cells of differing designs sourced from around the world, it was thought that any systematic errors due to the isotopic composition would be randomized; this was clearly not the case.

8 Comparison with Results from the CCT-K7 Key Comparison

In 2002–2003, the International Bureau of Weights and Measures (BIPM) coordinated an international comparison of WTP cell realizations, CCT-K7. Twenty NMIs sent a WTP cell, measured with respect to their own national reference cell(s), to the BIPM. In October 2002, cell 4-75 was measured with respect to the NMIA-2002 ensemble [11], sent to the BIPM, compared [7] with cells from 20 other laboratories participating in the CCT-K7 intercomparison, and returned to NMIA where it was again measured with respect to the NMIA-2002 ensemble [12]. The temperature of the NMIA-2002 ensemble with respect to the CCT-K7 key-comparison-reference-value (KCRV) was determined as $T_{\mathrm{NMI2002}} - T_{\mathrm{KCRV}} = (-51 \pm 72)\,\mu\mathrm{K}$ (at $k = 2$).

However, the CCT-K7 identified a significant ambiguity in the definition of the kelvin: two groups of laboratories were identified by the comparison, with an "upper" group of three laboratories lying nearly 100 µK above a "lower" group of 18 laboratories. This was identified as arising from an ambiguity in the definition of the isotopic concentration of the water used to define the SI unit of temperature. Two laboratories in the "upper" group had measured the isotope concentrations in the cells before sealing and had applied corrections to the temperatures, and the third measured the isotopic concentration to be close to VSMOW after the comparison. A proposal to amend the SI text to clarify the definition, CCT-T1 (2005), was submitted by the Consultative Committee for Thermometry (CCT) to the International Committee for Weights and Measures (CIPM) and approved in 2006. Although the comparison data showed evidence of two populations of cells, for the purposes of international comparability, a KCRV for this comparison was chosen as the simple mean of all results. The uncertainty in the value of $T_{\mathrm{NMI2002}} - T_{\mathrm{KCRV}} = (-51 \pm 72)\,\mu\mathrm{K}$ (at $k = 2$) was dominated by the measured stability of the transfer cell 4-75 while at BIPM, and so is uncorrelated with the difference $T_{\mathrm{NMI2006}} - T_{\mathrm{NMI2002}} = (107 \pm 31)\,\mu\mathrm{K}$ (95 % C.L.) obtained in the present study. We thus take the difference and the quadrature sum of these uncertainties to obtain $T_{\mathrm{NMI2006}} - T_{\mathrm{KCRV}} = (+56 \pm 78)\,\mu\mathrm{K}$ (95 % C.L.). In the CCT-K7 report, three laboratories other than the "upper group" had isotope data for their cells but had not applied corrections. When these three additional labs (making six in total) used their isotope-corrected data, the estimated difference between the "upper" population of laboratories applying isotopic corrections and the KCRV was $T_{\mathrm{SMOW,KCRV}} - T_{\mathrm{KCRV}} = (+73 \pm 30)\,\mu\mathrm{K}$ (at $k = 2$). The "NMI-2006 ensemble" measurements presented in this article are consistent with the cell ensemble belonging to the "upper" population of cells in the CCT-K7.

9 Conclusions

In accordance with the CIPM change to the definition of the kelvin [13], we have established a new ensemble of triple-point-of-water cells, from a variety of sources, for which isotopic correction data are available and have been applied. The measured variance of the new ensemble is much lower than that of NMIA's previous cell ensembles, with a mean value of $(107 \pm 31)\,\mu\text{K}$ (95 % C.L.) higher, consistent with the findings of the CCT-K7 report. NMIA has now adopted this new ensemble as defining the magnitude of the kelvin in Australia.

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