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CCT-K9.4

Freezing point of zinc fixed-point cells

KEY COMPARISON

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CCT-K9.4: Comparison of the freezing point of zinc fixed-point cells

Final Report

S. Dedyulin[1], A. Peruzzi[1], R. Veltcheva[2], J. Pearce[2]

[1]National Research Council of Canada, Ottawa, Canada
[2]National Physical Laboratory, Teddington, United Kingdom

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Contents

1 Organization of the comparison

1.1 Introduction

This comparison was initiated by National Physical Laboratory (NPL, UK) and was designed to compare the realization of the ITS-90 at the freezing point of zinc, 692.677 K (Zn FP) through a) the calibration of two SPRTs and b) independent measurements of a transfer open zinc cell. Each participant was to use two transfer standard SPRTs to measure two Zn fixed point cells, namely their own national standard cell and a transfer standard cell (see Appendix1: Technical Protocol). The primary objective was to link NPL Zn realization to CCT-K9 [1].

1.2 Participants and roles

Table 1 List of participant laboratories, contact persons and email addresses.

Participant	Contact Person	Contact Email
National Research Council of Canada (NRC, Canada, pilot)	Sergey Dedyulin Andrea Peruzzi	Sergey.Dedyuin@nrc-cnrc.gc.ca Andrea.Peruzzi@nrc-cnrc.gc.ca
National Physical Laboratory (NPL, UK)	Radka Veltcheva Jonathan Pearce	radka.veltcheva@npl.co.uk jonathan.pearce@npl.co.uk

1.3 Timeline

Table 2 Timeline of CCT-K9.4 bilateral comparison.

Action	Date
Final version of the CCT-K9.4 Technical Protocol is approved by all participants	Aug 4, 2023
NPL measurements of two transfer-standard SPRTs in Zn NPL national reference and NPL transfer standard cells	Jan-Feb, 2024
NRC measurements of two transfer-standard SPRTs in Zn NRC national reference and NPL transfer standard cells	Mar-Apr, 2024
NPL return measurements of one transfer-standard SPRT* in Zn NPL national reference and NPL transfer standard cells	Sep, 2024
NRC analyzes the data and prepares draft A of the report	May-Jun, 2025

*One of the transfer-standard SPRTs became unstable, most likely during the shipment to NRC. See Section 2.5 Stability of the transfer SPRTs for details.

2 Experimental details

2.1 Equipment used

Table 3 Equipment used by participants in CCT-K9.4 comparison.

Equipment	Model and serial number	Relevant parameters / notes
	NPL	
Zn transfer standard cell	NPL-made, s/n Zn 5/15	"Open" cell, 17.4 cm immersion*
Transfer SPRT 1	Isotech 670 SQ, s/n 312	Failed CCT-K9 stability criteria, see Section 2.5
Transfer SPRT 2	Isotech 670 SQ, s/n 583	Failed NRC stability criteria, see Section 2.5
Zn national reference cell	NPL-made, s/n Zn 10/09	"Open" cell, 17.4 cm immersion*
Triple point of water cell	Isotech B1150s/n 2028Q	
Resistance bridge	ASL F900, s/n 009340/02	1 mA, 0.5 Hz bandwidth
Zn furnaces	Fluke model 9114, s/n B06189 and A63118	Three zone
Standard resistor**	Tinsley 5685A, 100 Ω, 222106	temp sensitivity coefficient 1.5e-4 Ω/K
	NRC	
Zn reference cell in CCT-K9	NRC-made, s/n Zn-6	"Open" cell, 26.5 cm immersion*
Triple-point-of-water cell	Jarrett, s/nB11-2053	
Resistance bridge	MI 6020T-P DC bridge, s/n 1104565	1 mA, 10 s reversal
Zn furnace(s)	NRC-made	DC, single zone
Standard resistor***	Tinsley 5685A 100 Ω, s/n 262464	temp sensitivity coefficient 1.53e-4 Ω/K

** from the bottom of the thermal well to the surface of the metal in the crucible*
*** in the Fluke 7108 oil bath, stability of 20 mK monitored by a PRT*
**** in the Guildline oil bath model 9732VT, better than 10 mK temperature stability monitored by an SPRT*

2.2 Zn measurements at NRC

The freezing points of both cells – the NRC national reference in CCT-K9, Zn-6, and the transfer standard cell, Zn 5/15 – were realized simultaneously three times in two identical single-zone furnaces. Since both cells were "open" cells, a DH Instruments PPC3 pressure controller was used to control the pressure at 101.325 kPa throughout the freezes. Both cells were held 3.5 °C above the freezing point overnight prior to each measurement. To initiate the freeze, the furnace temperature was set 2.5 °C below the freezing point and the cooling process was monitored using the transfer standard SPRT in one of the cells (chosen randomly) until the recalescence was observed. At that point, the room-temperature quartz tubes were inserted for 2 minutes in both cells to initiate the inner solid interface and the furnace temperatures were set to 0.5 °C and 0.1 °C below the freezing point for the NRC cell Zn-6 and the NPL transfer standard cell, respectively. The temperature settings were selected to produce the length of the freezing plateau of approximately 10 hours in both cases. Each time, the temperature difference was measured at the beginning of the freezing plateau (approximately 1 h after the recalescence) using transfer standard SPRT. The same furnace setting was used for measuring both the temperature difference between the two cells and the immersion profiles.

2.3 Zn measurements at NPL

The national standard zinc cell serial number Zn10/09 and the transfer cell serial number Zn5/15 were manufactured by NPL in 2009 and 2015 respectively. Both cells are open design, filled with pure argon gas and the pressure inside controlled at 101.325 kPa.

Realisation of the freezing point of zinc by both transfer cell Zn 5/15 and national standard cell Zn 10/09 was done simultaneously. They were placed each into three zone furnaces and set to melt at temperature about 5 °C higher than melting temperature of zinc. The cells were held molten over night. To initiate freezing process the furnaces were set to about 0.2 °C below the freezing point and cooling process monitored by a SPRT. Once the recalescence is observed, the freeze was initiated by inserting a metal rod at room temperature into thermowell for about 1 minute followed by a SPRT (at room temperature). The cells were compared using three SPRTs in three different realisations. The measurement sequence in each cell starts with a SPRT and finishes with the same SPRT, so the range of the plateau can be evaluated.
Immersion profile of each of the cells is measured by each of the SPRTs.

2.4 Stability of NRC CCT-K9 reference cell

The NRC national reference in CCT-K9 was NRC-made Zn-6 open fixed-point cell. The design of the cell is identical to the Sn fixed-point cell described in [2]. The cell was filled in Feb, 2008 using 6N purity zinc (Johnson Matthey Lot # M9073 (0.3 kg) + Honeywell Lot # M18077 (1.0 kg)).
Prior to the CCT-K9 comparison, in Oct, 2012, Zn-6 was compared to a newly-filled Zn-7 cell (5N Plus Trail Inc., 7N purity Zn, Lot #137290, 1.3 kg) of a similar design:
$T_{\mathrm{NRC}}^{\mathrm{K9.2012}} - T_{\mathrm{Zn\text{-}7}}^{2012} = 0.290$ mK (1 measurement) [3]
Leading up to the CCT-K9.4 comparison, the same measurements were repeated in 2024:
$T_{\mathrm{NRC}}^{\mathrm{K9.2024}} - T_{\mathrm{Zn\text{-}7}}^{2024} = -0.379$ mK
$\sigma\left(T_{\mathrm{NRC}}^{\mathrm{K9.2024}} - T_{\mathrm{Zn\text{-}7}}^{2024}\right) = 0.009$ mK (3 measurements) [4]
The uncertainty budget for 2024 comparison of two zinc cells is provided in Appendix 3: Uncertainty budgets. For the lack of information, we used the same uncertainty for the comparison of two zinc cells in 2012 in Section 3 Analysis of the results using transfer fixed point cell.
Using the measured temperature difference between NRC K9 reference cell and Zn-7 cell measured in 2012 and in 2024, one can estimate the drift in NRC K9 reference cell since CCT-K9 comparison (for linking purposes):
$T_{\mathrm{NRC}}^{\mathrm{K9.2024}} - T_{\mathrm{NRC}}^{\mathrm{K9.2012}} = \left(T_{\mathrm{NRC}}^{\mathrm{K9.2024}} - T_{\mathrm{Zn\text{-}7}}^{2024}\right) - \left(T_{\mathrm{NRC}}^{\mathrm{K9.2012}} - T_{\mathrm{Zn\text{-}7}}^{2012}\right) = -0.669$ mK
In the calculation above, we have assumed that Zn-7 cell remained stable, while NRC K9 reference cell has drifted downwards. The opposite assumption, namely, that Zn-7 cell drifted

upwards while NRC K9 reference cell remained stable, was discarded for the following three reasons:
1) NRC K9 reference cell has visually deteriorated (Figure 1) – there are multiple cracks in the graphite envelope and in one spot the quartz envelope has changed color to milky white, potentially, due to contamination (the temperature appears too low for devitrification). This mode of failure of Zn cells, although not described in the literature, appears to be quite common as we have learned from conversations with our colleagues at the sidelines of TEMPMEKO 2025 conference. The reasons for the failure are currently unknown.

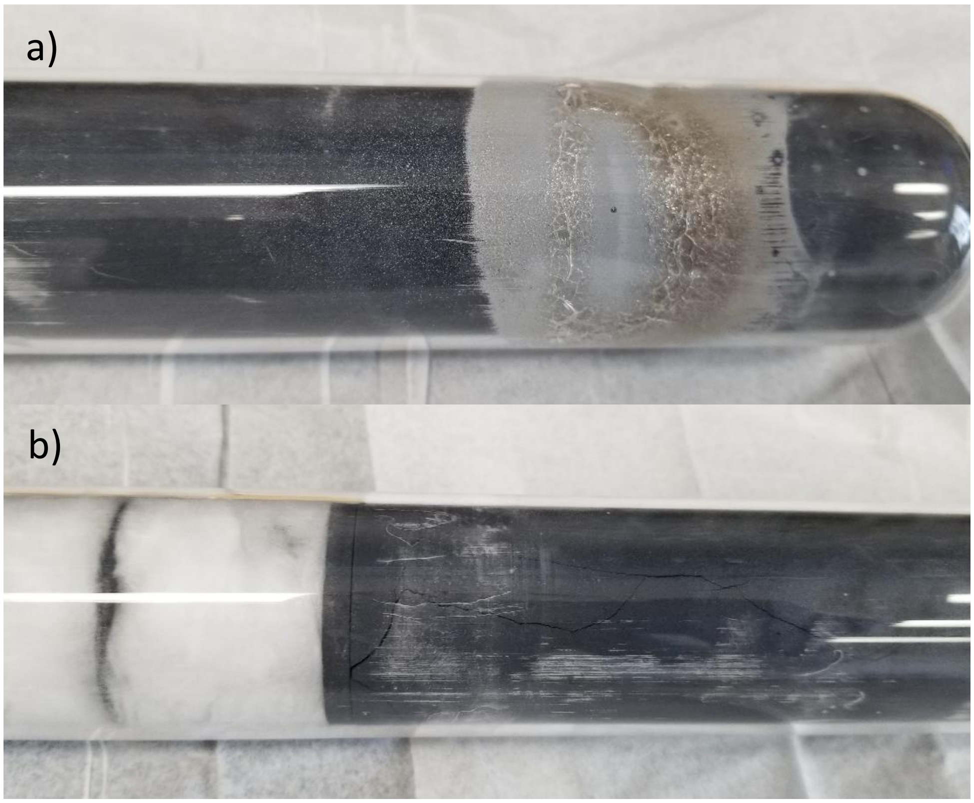

Figure 1. Picture of the NRC K9 Zn reference cell showing damage to the quartz envelope (a) and cracks in the graphite crucible (b).

2) Unlike NRC K9 reference cell, Zn-7 cell was not used since 2012 and stayed at room temperature, there was no signs of deterioration present in NRC K9 reference cell.
3) Zn-7 cell realized the temperature higher than NRC K9 reference cell when the two cells were intercompared in 2024 and most of the impurities lower the melting / freezing point.

2.5 Stability of the transfer SPRTs associated with the travel

Upon receiving two transfer SPRTs from NPL, NRC measured them in the triple point of water and then performed a customary 480 °C anneal overnight in the vertical furnace as per its quality procedure. If the difference between the SPRT's resistances measured at the TPW before and after the 480 °C soak was larger than the equivalent of 0.2 mK, the procedure was repeated until either the difference was smaller than 0.2 mK or the SPRT had been subjected to five overnight soaks. The results of the 480 °C soaks are shown in Table 4. It was concluded that transfer SPRT 2 became unstable during the shipment to NRC and it was excluded from further measurements after communicating with NPL. The transfer SPRT 1 (Table 3) was used in the comparison.

Table 4 Results of an overnight 480 °C anneal of transfer SPRTs: Isotech s/n 312 (SPRT 1) and Isotech s/n 583 (SPRT 2). Shown in brackets is the change from the previous measurement in mK.

Date	$R_{\mathrm{TPW}}^{\mathrm{SPRT\ 1}}$ **/ Ω (ΔT$_{\mathrm{prev}}$ / mK)**	$R_{\mathrm{TPW}}^{\mathrm{SPRT\ 2}}$ **/ Ω (ΔT$_{\mathrm{prev}}$ / mK)**
24/03/11 (as received)	25.3256552	25.7456792
24/03/12	25.3256954 (0.402)	25.7457664 (0.872)
24/03/13	25.3257145 (0.191)	25.7458346 (0.682)
24/03/14		25.7459235 (0.889)
24/03/15		25.7460562 (1.326)
24/03/18 (over weekend)		25.7464714 (4.152)

Upon receiving the return measurements results from NPL, the CCT-K9 cut-off criteria were used to ensure that uncertainty associated with the travel, handling, or stability of transfer SPRT did not dominate the standard uncertainty in measured temperature difference [1]:

$$\frac{\left|\overline{W}(\mathrm{Zn})_{\text{NPL, SPRT, Post-NRC}}-\overline{W}(\mathrm{Zn})_{\text{NPL, SPRT, Pre-NRC}}\right|}{\left({dW_r}/{dT}\right)\sqrt{u_R\left[\overline{W}(\mathrm{Zn})_{\text{NPL, SPRT, Post-NRC}}\right]^2+u_R\left[\overline{W}(\mathrm{Zn})_{\text{NPL, SPRT, Pre-NRC}}\right]^2}} > t_{0.95,\nu_{\mathrm{eff}}} \text{ (Cut-off criteria 1)}$$

$$u(C_{\mathrm{SPRT}}) > \frac{\sqrt{u(\Delta T_{\mathrm{NPL}})^2-u(C_{\mathrm{SPRT}})^2}}{3} \text{ (Cut-off criteria 2)}$$

Where C_{SPRT} is a term used to account for uncertainty associated with the travel, handling or stability of SPRT and is taken to have a value of $C_{\mathrm{SPRT}}=0$ and a standard uncertainty, $u(C_{\mathrm{SPRT}})$, of

$$u(C_{\mathrm{SPRT}})=\frac{\left|\overline{W}(\mathrm{Zn})_{\text{NPL, SPRT, Post-NRC}}-\overline{W}(\mathrm{Zn})_{\text{NPL, SPRT, Pre-NRC}}\right|}{\left({dW_r}/{dT}\right)\sqrt{12}}$$

It was found the both inequalities were fulfilled for the remaining transfer SPRT 1 meaning that the statistical agreement between SPRT's resistance ratios before and after its travel to NRC is poor. Therefore, in the analysis of the results of the comparison, the measurements of Zn transfer fixed-point cell (with the transfer SPRT 1) were used instead as described in the following section.

3 Analysis of the results using transfer fixed point cell

Since the stability of transfer-standard SPRTs was insufficient (see Section 2.5 Stability of the transfer SPRTs) we used the measurements of a Zn transfer fixed-point cell in our analysis of CCT-K9.4 results.

First NPL calibrated its transfer cell (Zn 5/15) against its national reference cell (Zn 10/09) using the SPRT (from NPL report, Appendix 2: Participants' reports on the calibration of the transfer cells):

$W_{\text{NPL1}}^{\text{Transf}} = 2.568554474$
$W_{\text{NPL1}}^{\text{NatRef}} = 2.568553820$
$T_{\text{NPL1}}^{\text{Transf}} - T_{\text{NPL1}}^{\text{NatRef}} = \frac{(W_{\text{NPL1}}^{\text{Transf}} - W_{\text{NPL1}}^{\text{NatRef}})}{\frac{dW_{\text{r}}}{dT}} = 0.187 \text{ mK}$
$U(T_{\text{NPL1}}^{\text{Transf}} - T_{\text{NPL1}}^{\text{NatRef}}) = 0.428$ mK (from NPL report, Appendix 3: Uncertainty budgets)

Then NRC calibrated NPL transfer cell (Zn 5/15) against NRC national reference cell (Zn6) using the SPRT (from NRC report, Appendix 2: Participants' reports on the calibration of the transfer cells):
$W_{\text{NRC}}^{\text{Transf}} = 2.568551900$
$W_{\text{NRC}}^{K9.2024} = 2.568551794$
$T_{\text{NPL}}^{\text{Transf}} - T_{\text{NRC}}^{\text{K9.2024}} = \frac{(W_{\text{NRC}}^{\text{Transf}} - W_{\text{NRC}}^{\text{K9.2024}})}{\frac{dW_{\text{r}}}{dT}} = 0.030 \text{ mK}$
$U(T_{\text{NPL}}^{\text{Transf}} - T_{\text{NRC}}^{\text{K9.2024}}) = 0.777$ mK (from NRC report, Appendix 3: Uncertainty budgets)

Finally, NPL re-calibrated its transfer cell (Zn 5/15) against its national reference cell (Zn 10/09) using the SPRT (from NPL report, Appendix 2: Participants' reports on the calibration of the transfer cells):
$W_{\text{NPL2}}^{\text{Transf}} = 2.568552784$
$W_{\text{NPL2}}^{\text{NatRef}} = 2.568551634$
$T_{\text{NPL2}}^{\text{Transf}} - T_{\text{NPL2}}^{\text{NatRef}} = \frac{(W_{\text{NPL2}}^{\text{Transf}} - W_{\text{NPL2}}^{\text{NatRef}})}{\frac{dW_{\text{r}}}{dT}} = 0.329 \text{ mK}$
$U(T_{\text{NPL2}}^{\text{Transf}} - T_{\text{NPL2}}^{\text{NatRef}}) = 0.428$ mK (from NPL report, Appendix 3: Uncertainty budgets)

The difference between transfer cell and national reference at NPL is obtained by averaging the results obtained before and after sending the transfer cell to NRC:
$T_{\text{NPL}}^{\text{Transf}} - T_{\text{NPL}}^{\text{NatRef}} = \frac{(T_{\text{NPL1}}^{\text{Transf}} - T_{\text{NPL1}}^{\text{NatRef}}) + (T_{\text{NPL2}}^{\text{Transf}} - T_{\text{NPL2}}^{\text{NatRef}})}{2} = \frac{0.187 + 0.329}{2} = 0.258 \text{ mK}$
$U(T_{\text{NPL2}}^{\text{Transf}} - T_{\text{NPL2}}^{\text{NatRef}}) = \frac{1}{\sqrt{2}} * 0.428 = 0.303 \text{ mK}$
$D_{2024} = T_{\text{NPL}}^{\text{NatRef}} - T_{\text{NRC}}^{\text{K9.2024}} = (T_{\text{NPL}}^{\text{Transf}} - T_{\text{NRC}}^{\text{K9.2024}}) - (T_{\text{NPL}}^{\text{Transf}} - T_{\text{NPL}}^{\text{NatRef}}) = 0.030 - 0.258 = -0.228 \text{ mK}$

Bilateral equivalence between NPL and NRC K9 reference in 2012:
$D_{2012} = T_{\text{NPL}}^{\text{NatRef}} - T_{\text{NRC}}^{\text{K9.2012}} = (T_{\text{NPL}}^{\text{NatRef}} - T_{\text{NRC}}^{\text{K9.2024}}) + (T_{\text{NRC}}^{\text{K9.2024}} - T_{\text{NRC}}^{\text{K9.2012}}) = -0.228 - 0.669 = -0.897 \text{ mK}$

Difference between NPL and KCRV K9:
In K9, NRC temperature difference from KCRV was: $\Delta T_{\text{NRC-KCRV}}^{2012} = 0.30$ mK (table 5.2, CCT-K9 report [1])
$\Delta T_{\text{NPL-KCRV}}^{2024} = (T_{\text{NPL}}^{\text{NatRef}} - T_{\text{NRC}}^{\text{K9.2012}}) + \Delta T_{\text{NRC-KCRV}}^{2012} = -0.90 + 0.30 = -0.60 \text{ mK}$

Uncertainty bilateral equivalence:

$$U(\Delta T_{\text{NPL-KCRV}}^{2024}) =$$
$$\sqrt{\left(\left(U\left(T_{\text{NPL}}^{\text{Transf}} - T_{\text{NPL}}^{\text{NatRef}}\right)\right)^2 + (U\left(T_{\text{NRC}}^{\text{Transf}} - T_{\text{NRC}}^{\text{K9.2024}}\right))^2 + (U_{\text{NRC}}^{\text{K9.2024/Zn-7}})^2 + (U_{\text{NRC}}^{\text{K9.2012/Zn-7}})^2 + U(\Delta T_{\text{NRC-KCRV}}^{2012})\right)} =$$
$$\sqrt{(0.303^2 + 0.777^2 + 0.567^2 + 0.567^2 + 0.75^2)} = 1.38\ \text{mK}$$

References

[1] Tobias Herman et al "CCT Key Comparison 9 Final Report: ITS-90 SPRT calibration from the Ar TP to the Zn FP" *Metrologia* **60** (2023) 03001.
[2] Bernd Fellmuth and Kenneth D Hill "Estimating the influence of impurities on the freezing point of tin" *Metrologia* 43 (2006) 71.
[3] Ken Hill, NRC Laboratory Notebook #37, p. 25
[4] Sergey Dedyulin, NRC Laboratory Notebook #17, pp. 1--5

Appendix1: Technical Protocol

CCT-K9.4

Comparison of the freezing point of zinc fixed-point cells using SPRTs

Technical protocol

Sergey Dedyulin (NRC), Andrea Peruzzi (NRC), Jonathan Pearce (NPL)

Introduction

This comparison is initiated by NPL and is designed to compare the realization of the ITS-90 at the freezing point of zinc, 692.677 K (Zn FP) through a) the calibration of SPRTs and b) independent measurements of a transfer open zinc cell. Each participant will use two transfer standard SPRTs to measure two Zn fixed point cells, namely their own national standard cell and a transfer standard cell. The transfer standards used will provided by NPL, and will be two long-stem SPRTs and an open Zn fixed-point cell.

The reference documents followed in drawing up the technical protocol are a) "Comparison protocol template 20170505.docx" supplied by NRC and b) the CCT-K9 intercomparison protocol.

Participants

Pilot: NRC, Sergey Dedyulin, sergey.dedyulin@nrc-cnrc.gc.ca and Andrea Peruzzi, andrea.peruzzi@nrc-cnrc.gc.ca

Participant: NPL, Jonathan Pearce, jonathan.pearce@npl.co.uk

Projected Timeline:

Protocol agreement	31 August, 2023
Transfer standards sent to NRC	30 January, 2024
Transfer standards returned to NPL	30 April, 2024
Draft A report completed	31 August 2024

Measurement instructions

The calibration of two transfer SPRTs by NPL and NRC is to be performed using the local national standard Zn fixed-point cells and a transfer standard Zn fixed-point cell of the open design according to an appropriate procedure used by each participating NMI.

Participants will supply the following as appropriate:

- 2 SPRTs calibrated at FP Zn
    - NPL will select their own SPRTs based on their own criteria for suitability and transport them to and from NRC by hand.
    - In case of failure of the SPRTs during the measurements at one NMI, the other NMI shall be notified and a way forward shall be discussed.
    - The two NPL SPRTs must be calibrated by NPL before measurements are made at NRC and then again on return from NRC
- Fixed point cells
    - NPL will select a transfer open Zn fixed-point cell based on their own criteria for suitability and transport it to and from NRC

    - NPL and NRC will select an local open Zn fixed-point cell based on their own criteria for suitability
    - In case of failure of the transfer fixed-point cell during the measurements at one NMI, the other NMI shall be notified and a way forward shall be discussed.
- Calibration results supplied in terms of *W* with all corrections applied by each NMI such that the W values are equivalent to the ITS-90 assigned temperature values for 0 mA. Uncertainties, *u*(*W*), may be specific to each SPRT or a nominal uncertainty may be given for both SPRTs.
- Immersion profiles in all Zn fixed-point cells (local and transfer) must be measured at least once, with measuring current of 0 mA and heights above the thermometer well at the NMI's discretion but to include 5 mm, 10 mm, 20 mm, 40 mm.
- Care must be taken to measure the resistance of each SPRT at the triple point of water (TPW) at various points in the measurements, as outlined in Appendix A.
- Uncertainty budget compliant with CCT CMC requirements that includes degrees of freedom associated with each component
    - A suggested fixed-point cell uncertainty budget is given in Appendix B
        - Sources of uncertainty may be added or deleted as needed
        - NMIs may choose to supply their own uncertainty budget (CCT CMC compliant) that includes degrees of freedom for each source of uncertainty
        - Please identify which components of the uncertainty budget are associated with random effects in *W*(FP) and which are associated with systematic effects in *W*(FP) within this comparison.
        - Heat flux (immersion) profile for each fixed-point cell using the SPRTs of this comparison
            - *R*(FP), 0 mA and corresponding immersion depth (sensor midpoint), in units of cm
        - All results and required information will be emailed to NRC
            - Andrea Peruzzi, andrea.peruzzi@nrc-cnrc.gc.ca
            - Sergey Dedyulin, Sergey.Dedyulin@nrc-cnrc.gc.ca

Organizational aspects

In case of an unexpected delay at either NMI, the other shall be informed and an updated schedule agreed on.

The fixed-point cell and SPRTs shall travel under an ATA carnet to be arranged by NPL.

NPL shall be responsible for the transport and customs costs associated with the travelling fixed-point cell and SPRTs.

In the event of damage to the fixed-point cell or SPRTs, NPL will endeavour to supply replacements.

Communication flows

Any measurement delays at either NMI will be promptly communicated to the other.
NPL shall communicate the measurement results to NRC by the due date. If either NMI cannot comply with the due dates they will discuss with the other.

Reporting the results

The measurement results shall be reported using the template in Appendix A.
The measurement uncertainties shall be reported using the template in Appendix B.

KCRV and linkage mechanism

The intercomparison will link to the CCT-K9 KCRV via NRC. If the SPRTs do not drift significantly, the SPRTs will be used for the linkage (the transfer cell represents insurance against this).
The intercomparison is a direct one between NRC and NPL.

Appendix 2: Participants' reports on the calibration of the transfer cells

Measurement report worksheet (active Excel Spreadsheet)

For measurements at NPL

Before sending SPRTs and cell to NRC

	SPRT 1			SPRT 2		
Initial R(TPW)	25.325 740 10			25.7456179		
R(TPW) after annealing	25.325 743 80			25.7456359		
	SPRT 1	u(W), mK	Number of equilibria realized	SPRT 2	u(W), mK	Number of equilibria realized
W(Zn NPL national standard)	2.568 553 820	0.59	3	2.568 648 168	0.59	3
W(Zn transfer standard)	2.568 554 474	0.59	3	2.568 649 187	0.59	3
Final R(TPW)	25.325 765 87			25.745 745 88		

On return of SPRTs and cell from NRC

	SPRT 1			SPRT 2		
On receipt R(TPW)	25.325 647 50					
R(TPW) after annealing	25.325 655 50					

SPRT 583 is not used due to the large drift experienced at NRC

	SPRT 1	u(W), mK	Number of equilibria realized	SPRT 2	u(W), mK	Number of equilibria realized
W(Zn NPL national standard)	2.568 551 634	0.61	3			
W(Zn transfer standard)	2.568 552 784	0.61	3			
Final R(TPW)	25.325 664 57					

Fixed-point cell information

	s/n	Immersion depth*, cm	Pressure, kPa
Zn NPL national standard	Zn 10/09	17.4	101.325
Zn transfer standard	Zn 5/15	17.4	101.325

*from the bottom of thermowell to the surface of the metal inside the crucible

	Resistance bridge model	Reference resistor model	Resistor enclosure stability, mK
Measurement system	F900	Tinsley 100 Ohms	20 mK

For measurements at NRC

On receipt of SPRTs from NPL

	SPRT 1	s/n 312		SPRT 2	s/n 583	
On receipt R(TPW)	25.325 673 10	11-Mar-24		25.745 697 45	11-Mar-24	
After anneal R (TPW)	25.325 732 50	13-Mar-24		25.746 489 65	18-Mar-24	

SPRT 583 was not used - didn't stabilize during anneal, see SD book 14, pp. 150-153

	SPRT 1	u(W), mK	Number of equilibria realized	SPRT 2	u(W), mK	Number of equilibria realized
W(Zn-6, Nat Stand in CCT-K9)	2.568 551 794	0.95	3			
W(Zn transfer standard)	2.568 551 900	0.95	3			
Final R(TPW)	25.325 686 10	27-Aug-24				

Fixed-point cell information

	s/n	Immersion depth*, cm	Pressure, kPa
Zn NRC national standard	Zn-6	26.5	101.325

*from the bottom of thermowell to the surface of the metal inside the crucible

	Resistance bridge model	Reference resistor model	Resistor enclosure stability, mK
Measurement system	MI 6020T-P	Tinsley 100 Ohms	10

Appendix 3: Uncertainty budgets

NPL: Uncertainty budget worksheet (active Excel Spreadsheet)

1. Calibration of a Zn freezing point cell by comparison with open reference cell

Component	Description	Standard uncertainty	Comment	Sensitivity coefficient	Uncertainty contribution ui in mK
A1	Repeatability of comparisons (0 mA)	0.07	s.d. of 9 meast's	1	0.023
B1	Uncertainty of ref (open) cell	0.205	from NPL ref cell budget	1	0.205
B2	Hydrostatic pressure correction	10 mm / √3		2.7 mK/m	0.016
B3	Argon pressure in ref cell	2.6 kPa / √3	incl. in cell calibration	4.3 E-8 K/Pa	0.000
B4	Perturbing heat exchanges	0.09 mK / √3	Immersion tests	1	0.052
B5	Self-heating extrapolation: bridge current ratio	2% of SHE (2 mK)	correlated	1	0.000
B6	Bridge linearity (differential)	0	correlated	1	0.000
B7	Temperature of Rs	0.02 °C / √3	for TC < 2 ppm/°C	0.74 mK/ppm	0.017
B8	AC/DC, frequency, etc	1.04E-5 / √3	correlated	11.4E3 mK	0.000
B9	Internal insulation leakage	not estimated	N/A		0.000
	Sub-total Type B				**0.213**
Combined uncertainty					**0.214**
Effective degrees of freedom					28088
Expanded uncertainty				k = 2	**0.428**

2. Calibration of an SPRT at the freezing point of Zn

0 mA

Component	Description	Standard uncertainty	Comment	Sensitivity coefficient	Uncertainty contribution ui in mK
	Zinc				
A1	Repeatability of readings, including extrap to 0 mA	0.523E-6 Ω	s.d.m of 20 meas'ts	11.4E3 mK/Ω	0.006
B1	Uncertainty of ref cell	0.214	from previous budget	1	0.214
B2	Hydrostatic pressure correction	10 mm / √3		2.7 mK/m	0.016
B3	Argon pressure in cell	2.6 kPa / √3	incl. in cell calibration	4.3 E-8 K/Pa	0.000
B4	Perturbing heat exchanges	0.09 mK / √3	Immersion tests	1	0.052
B5	Self-heating extrapolation: bridge current ratio	2% of SHE (2 mK)	0.5% in current ratio	1	0.023
B6	Bridge linearity	0.82 E-7 / √3		1140E3 mK	0.054
B7	Temperature of Rs	0.02 °C / √3	for TC < 2 ppm/°C	0.74 mK/ppm	0.017
B8	AC/DC, frequency, etc	1.04E-5 Ω/ √3	high - low freq check	11.4e3 mK/Ω	0.068
B9	Internal insulation leakage	not estimated			0.000
	Sub-total at FP Zn				**0.239**
	TPW				
A1	Repeatability of readings, and extrap to 0 mA	0.34 E-6 Ω	s.d.m of 20 meas'ts	10000 mK/Ω	0.003
B1	Uncertainty of TP cell	0.038	MS cell uncertainty		0.038
B2	Hydrostatic pressure correction	5 mm / √3	5 mm in head	0.73 mK/m	0.002
B3	Residual gas pressure	-	Included in B1		0.000
B4	Perturbing heat exchanges	0.01 mK / √3	from TPW cell budget	1	0.006
B5	Self-heating extrapolation: bridge current ratio	2% of SHE (2 mK)	0.5% in current ratio	1	0.023
B6	Bridge linearity	0.82 E-7 / √3		1e6 mK	0.047
B7	Temperature of Rs	0.02 °C / √3	for TC < 2ppm/°C	0.25 mK/ppm	0.006
B8	AC/DC, frequency, etc	1E-6 Ω / √3	high - low freq check	10000 mK/Ω	0.006
B9	Internal insulation leakage	not estimated			0.000
	Sub-total at 0.01 °C				**0.066**
B10	**Equivalent at FP Zn**	0.066		2.93	**0.193**
Combined uncertainty					**0.307**
Effective degrees of freedom* **					52505
Expanded uncertainty				*k* = 2	**0.615**

NRC: Uncertainty budget worksheet (active Excel Spreadsheet)

1. Calibration of a Zn freezing point cell by comparison with open reference cell NPL

Component	Description	Comment	Uncertainty contribution ui in mK
A1	Repeatability of comparisons (0 mA)	std. dev. of 3 meas	0.035
B1	Uncertainty of ref (open) cell	Chemical impurities + slope of the plateau	0.266
B2	Hydrostatic pressure correction		0.027
B3	Ar pressure in reference cell		0.006
B4	Perturbing heat exchanges	from imm tests, Zn5-15, first 5 cm	0.279
B5	Self-heating extrapolation: bridge current ratio		0.010
B6	Bridge linearity (differential)	correlated	0.000
B7	Reference reistor stability	correlated	0.000
B8	Internal insulation leakage	not estimated	0.000
	Sub-total Type B		**0.387**
Combined uncertainty			**0.388**
Effective degrees of freedom			
Expanded uncertainty			**0.777**

2. Calibration of an SPRT at the freezing point of Zn 0 mA

Type	Description	Comment	Uncertainty contribution in mK
	Zinc		
A1	Phase transition repeatability	std. dev. of 3 meas Zn 5-15 (largest)	0.031
B1	Uncertainty of ref (open) cell	Chemical impurities + slope of the plateau	0.266
B2	Hydrostatic pressure correction		0.027
B3	Ar pressure in reference cell		0.006
B4	Perturbing heat exchanges	from imm tests, Zn5-15, first 5 cm	0.279
B5	Self-heating extrapolation: bridge current ratio		0.010
B6	Bridge linearity (differential)	15 ppb manuf spec (ratio 1) /sqrt(3)	0.009
B7	Reference reistor stability	bath stability 10 mK, 1.53e-4 Ω/K / sqrt(3)	0.010
B8	Internal insulation leakage	not estimated	0.000
	Sub-total at FP Zn		**0.388**
	TPW		
A1	Phase transition repeatability	std. dev. of the TPW meas corresp. To Zn meas above	0.091
B1	Uncertainty of ref (open) cell	cal TPW cell against Nat Ref, from Peruzzi_2022	0.018
B2	Hydrostatic pressure correction	Water level+position of SPRT, from Peruzzi_2022	0.003
B3	Residual gas pressure	included in B1	0.000
B4	Perturbing heat exchanges	From Peruzzi_2022	0.008
B5	Self-heating extrapolation: bridge current ratio	From Peruzzi_2022	0.007
B6	Bridge linearity (differential)	20 ppb manuf spec (ratio 0.1) / sqrt (3)	0.012
B7	Reference reistor stability	bath stability 10 mK, 1.53e-4 Ω/K / sqrt(3)	0.008
B8	Internal insulation leakage	not estimated	0.000
	Sub-total at 0.01 °C		**0.094**
	Equivalent at FP Zn		**0.277**
Combined uncertainty			**0.477**
Effective degrees of freedom* **			
Expanded uncertainty (*k* = 2)			**0.953**

3. Calibration of a Zn freezing point cell by comparison with open reference cell **NRC Zn6/Zn7**

Component	Description	Comment	Uncertainty contribution ui in mK
A1	Repeatability of comparisons (0 mA)	std. dev. of 3 meas	0.009
B1	Uncertainty of ref (open) cell	Chemical impurities + slope of the plateau	0.266
B2	Hydrostatic pressure correction		0.027
B3	Ar pressure in reference cell		0.006
B4	Perturbing heat exchanges	from imm tests, Zn5-15, first 5 cm	0.092
B5	Self-heating extrapolation: bridge current ratio		0.010
B6	Bridge linearity (differential)	correlated	0.000
B7	Reference reistor stability	correlated	0.000
B8	Internal insulation leakage	not estimated	0.000
	Sub-total Type B		**0.283**
Combined uncertainty			**0.283**
Effective degrees of freedom			
Expanded uncertainty			**0.567**

Appendix 4: Immersion profiles

As per measurement protocol (Appendix1: Technical Protocol), NPL and NRC provided an immersion profile for both national-reference and transfer-standard cell using transfer SPRT 1 (s/n 312). The immersion profiles were recorded with measuring current of 0 mA and included heights of 5 mm, 10 mm, 20 mm, 40 mm above the bottom of the thermometer well. At the NRC all profiles were measured from bottom to top using a linear translation stage with an integrated controller (Thorlabs LTS300). At NPL, thermometer was manually lifted from the bottom of the thermowell to 5 mm, 10 mm, 20 mm, 30 mm and 40 mm. Immersion profile was measured on withdraw and on insertion of the SPRT.

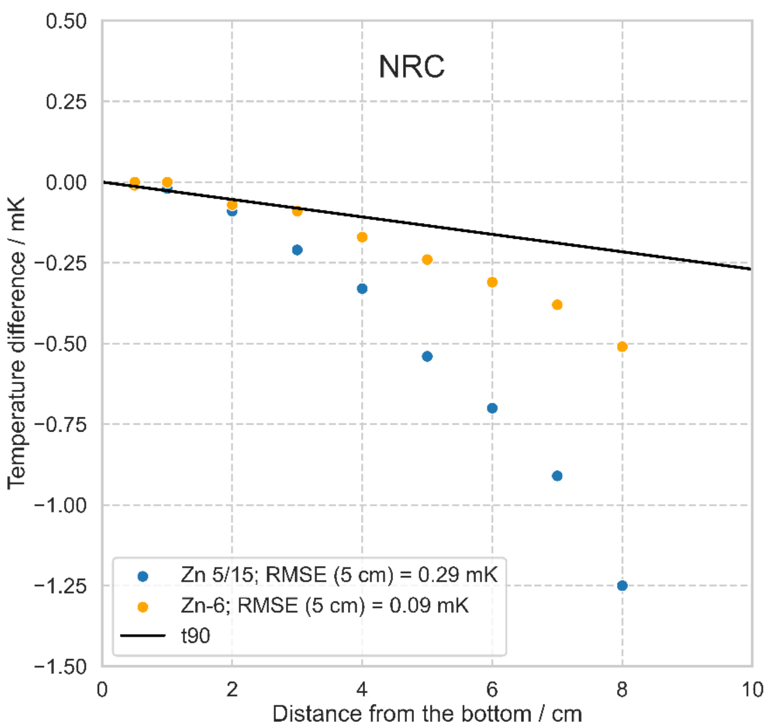

*Figure **Error! No text of specified style in document.** Immersion profile of NRC national reference in CCT-K9 (Zn-6) and Zn transfer standard cell (Zn 5/15) measured using transfer standard SPRT1 (s/n 312). Root mean square error values (RMSE) are shown in the legend.*

Table A4.1 Temperature differences plotted in Figure A4.1 with respect to the first measurement point at 0.5 cm from the bottom of the thermometer well.

Distance / cm	$\Delta T_{\mathrm{meas}}^{\mathrm{Zn\text{-}6}}$ **/ mK**	$\Delta T_{\mathrm{meas}}^{\mathrm{Zn\ 5/15}}$ **/ mK**	$\Delta T_{\mathrm{ITS\text{-}90}}$**/ mK**
0.5	0.00	0.00	0.00
1	0.00	-0.02	-0.01
2	-0.07	-0.09	-0.04
3	-0.09	-0.21	-0.07
4	-0.17	-0.33	-0.09
5	-0.24	-0.54	-0.12
6	-0.31	-0.70	-0.15
7	-0.38	-0.91	-0.18
8	-0.52	-1.25	-0.20

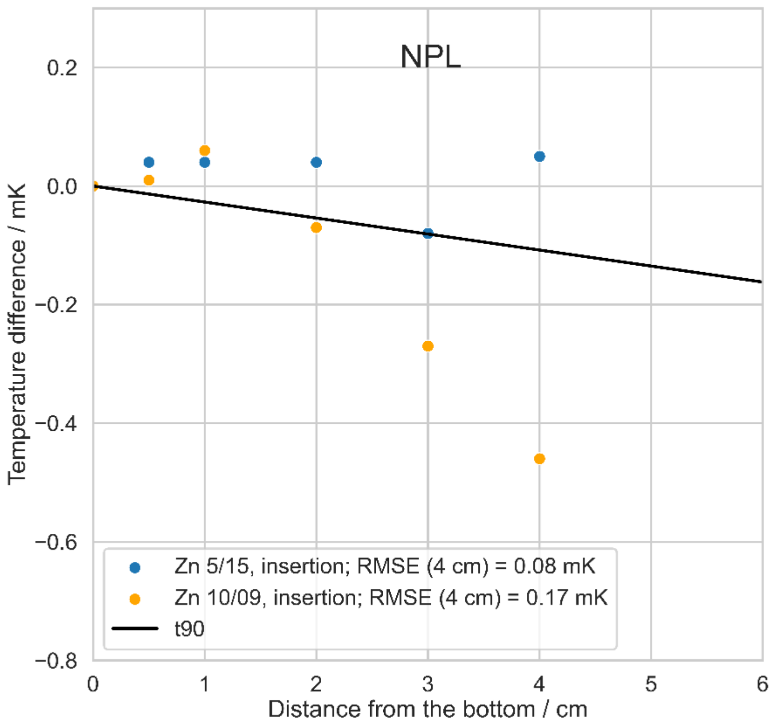

Figure A4.2 Immersion profile of NPL national reference cell (Zn 10/09) and Zn transfer standard cell (Zn 5/15) measured using transfer standard SPRT1 (s/n 312). Root mean square error values (RMSE) are shown in the legend.

Table A4.2 Temperature differences plotted in Figure A4.2 with respect to the measurement point at the bottom of the thermometer well.

Distance / cm	$\Delta T^{\mathrm{Zn\ 10/09}}_{\mathrm{meas}}$ **/ mK**	$\Delta T^{\mathrm{Zn\ 5/15}}_{\mathrm{meas}}$ **/ mK**	$\Delta T_{\mathrm{ITS\text{-}90}}$**/ mK**
4	-0.46	0.05	-0.11
3	-0.27	-0.08	-0.08
2	-0.07	0.04	-0.05
1	0.06	0.04	-0.03
0.5	0.01	0.04	-0.01
0	0.00	0.00	0.00