EURAMET-T.K9.1

Bilateral comparison of ITS-90 SPRT Calibration from the Hg TP to the Zn FP

Report (Final)

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Report to the CCT on the bilateral comparison EURAMET.T-K9.1

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1 Introduction

The EURAMET-T.K9.1 interlaboratory comparison links METROSERT to EURAMET-T.K9. The objective was to obtain the degree of equivalence for Metrosert with respect to the reference value of the CCT K9; at the triple point of Hg, the melting point of Ga, and the freezing points of Sn and Zn. The link is obtained via Justervesenet's (JV's) linkage to that value. The ultimate aim is to support Metrosert in a later CMC claim for their capabilities at the points in question.

The comparison was jointly planned by JV and Metrosert from early summer 2016, with a draft version of the protocol ready in early fall that year. The review process was somewhat delayed, but the approved protocol was ready in June 2017.

The protocol closely followed the protocol of the parent comparison, the EURAMET-T.K9, using an SPRT as the circulating instrument. The topology was quite simple with only two participants: the sequence of measurements were METROSERT – JV – METROSERT. The probe was hand carried between the labs.

1.1 Actual measurement schedule

Measurements were conducted from July to November, 2017. No significant issues were encountered during the measurements or transportation. Metrosert resoldered one of the wire terminals on the transfer standard, but this is not expected to affect the behaviour of the SPRT. However, the work did take longer than anticipated due to other obligations at the participating labs. The delay was not significant and should not impact the results in any way.

Table 1 The actual measurement schedule.

Stage	Started - ended	Report received
Metrosert, first set of measurements	12.07.2017 - 29.07.2017	28.08.2017
JV	30.08.2017 - 13.09.2017	12.10.2017
Metrosert, second set of measurements	07.11.2017 - 16.11.2017	08.12.2017

2 Participant data

JV		
Country:	Norway	
Institute (name/address):	Justervesenet (Norwegian Metrology Service) PO Box 170 2027 Kjeller Norway	
Persons involved:	Åge Andreas Falnes Olsen, coordinator	aao@justervesenet.no
	Reidun Anita Bergerud	rab@justervesenet.no
	Karsten Opel	kop@justervesenet.no

METROSERT		
Country:	Estonia	
Institute:	AS Metrosert Teaduspargi 8 12618 Tallinn Estonia	
Persons involved:	Toomas Kübarsepp	Toomas.Kubarsepp@metrosert.ee
	Kristjan Tammik	Kristjan.Tammik@metrosert.ee

3 Circulating instrument

The circulating instrument was an SPRT owned by Metrosert:

Model:	670 SQ Pt25
Manufacturer:	Isotech
Serial number:	052
Owner:	Metrosert

4 Equipment and conditions at the laboratories

Laboratory equipment at Metrosert:

Table 2 Measurement equipment at Metrosert.

Equipment	Manufacturer, model, type	serial no	Additional information	
Fixed point cells			*Depth (cm)*	*top*
Zn	Isotech, 492, metal	341464/2	15,3	Sealed
Sn	Isotech, 491, metal	341464/1	15,3	Sealed
Ga	Isotech, Ga232	ITL M 17401	20,2	Sealed
Hg	Fluke, 5900E, metal	000047	18,5	Sealed
TPW	Isotech, A11	J2027	28,5	Sealed
TPW	Isotech, B11	B11-320	26,5	Sealed
Resistance bridge			*Resolution*	*Linearity*
	MI, 6010T		10^{-9} of full scale	10^{-8}
Reference resistor			*Enclosure stability*	*Nominal R*
	Tinsley, 5685A		2 mK	25 Ω

Metrosert uses Isotech Isotower units to realise the freezing points of Sn and Zn, which are integrated systems comprising a furnace and a fixed point cell with preprogrammed ramp cycles to realise the fixed point plateaus. The cells are sealed with no pressure or atmospheric control. The Ga point is also realised using an automatic, fully integrated system. The triple point of Hg is realised with a traditional fixed point cell and a separate refrigeration unit.

Metrosert conducts all experiments in a temperature and humidity controlled laboratory, with room temperature of 22,0 °C ± 2 °C and relative humidity 40 %RH ± 10 %RH. The actual conditions are monitored and logged continuously. During the experiments for this comparison the values remained within the specified limits.

The resistance bridge resolution and linearity are quoted as ratio resolution and linearity, respectively. They correspond to temperature variations in the order of 1-2 µK.

Laboratory equipment at JV:

Table 3 Measurement equipment at JV. The resolution and linearity of the measurement bridge is expressed as relative numbers for the ratio value produced by the bridge.

Equipment	Manufacturer, model, type	serial no	Additional information	
Fixed point cells			*Depth (cm)*	*top*
Zn	Fluke, quartz	Zn-06077	16	Sealed
Sn	Fluke, quartz	Sn-05110	16	Sealed
Ga	Isotech, Automatic setup	Ga 442	20	Sealed
Hg	Fluke Hart, metal	Hg-07127	15	Sealed
TPW	Fluke Hart, 5901D-G	D-G1053	26.5	Sealed
Resistance bridge			*Resolution*	*Linearity*
	MI, 6010T		10^{-9} of full scale	10^{-8}
Reference resistor			*Enclosure stability*	*Nominal R*
	Tinsley, 5685A		5 mK	100 Ω

JV realises the Sn and Zn points using separate cells in a furnace chamber lined with an isothermal heatpipe liner to provide axially uniform temperature. The cells are sealed cells without any independent pressure or atmospheric control system. The Ga point is realised using a fully automatic, integrated system comprising a fixed point cell, a heater and appropriate control units. The triple point of Hg is realised with a fixed point cell and a separate refrigeration unit.

JV conducts all experiments in a temperature and humidity controlled laboratory, with room temperature of 22,0 °C ± 2 °C and relative humidity 40 %RH ± 10 %RH. The actual conditions are monitored and logged continuously. During the experiments for this comparison the values remained within the specified limits.

The resistance bridge resolution and linearity are quoted as ratio resolution and linearity, respectively. They correspond to temperature variations in the order of 1-2 µK.

The fixed point cells at JV and Metrosert are different, and at Zn and Sn from different vendors.

5 Measurement results

This section summarises the results reported by each participant. The main results is the corrected W value at each of the fixed points, which forms the basis for the calculation of the linkage (see section 5.4 for the corrections actually used, reported by each participant). Auxiliary results are the triple point of water measurements recorded by the participants, the immersion profiles measured in the fixed point cells, and the corrections applied to the measured resistance in each of the fixed points to account for self-heating and hydrostatic pressure in the cells.

5.1 SPRT resistance ratio at the fixed points

Table 4: Reported resistance ratios, along with standard uncertainty. The difference C in W after and before the measurements at JV, W(round2) – W(round1). The reported uncertainty (in mK as requested in the protocol) was converted to W using the sensitivity of the SPRT reference function (see Section 6).

Fixed point	$\boldsymbol{W}$ **(round 1)**	$\boldsymbol{W}$ **(round 2)**	$\boldsymbol{u}$ **(**$\boldsymbol{k}$**=1, mK)**	$\boldsymbol{C}$ (in $\boldsymbol{W}$)	$\boldsymbol{u_W}$ **(**$\boldsymbol{k}$**=1)**
Metrosert					
Hg	0,8441745	0,8441744	1,5	1E-07	6,1E-06
Ga	1,1181102	1,1181101	0,6	1E-07	2,4E-06
Sn	1,8925829	1,8925879	2,0	-5E-06	7,4E-06
Zn	2,5685412	2,5685337	2,6	7,5E-06	9,1E-06
JV					
Hg	0,8441732	-	0,30	-	1,2E-06
Ga	1,1181116	-	0,29	-	1,1E-06
Sn	1,8925863	-	0,40	-	1,5E-06
Zn	2,5685349	-	0,64	-	2,2E-06

The figures below summarises the tabulated values. Error bars represent the standard uncertainty reported by the participants. The reported uncertainties were in mK, and in order to convert to W Equation 6-1 is used (see Section 6.1).

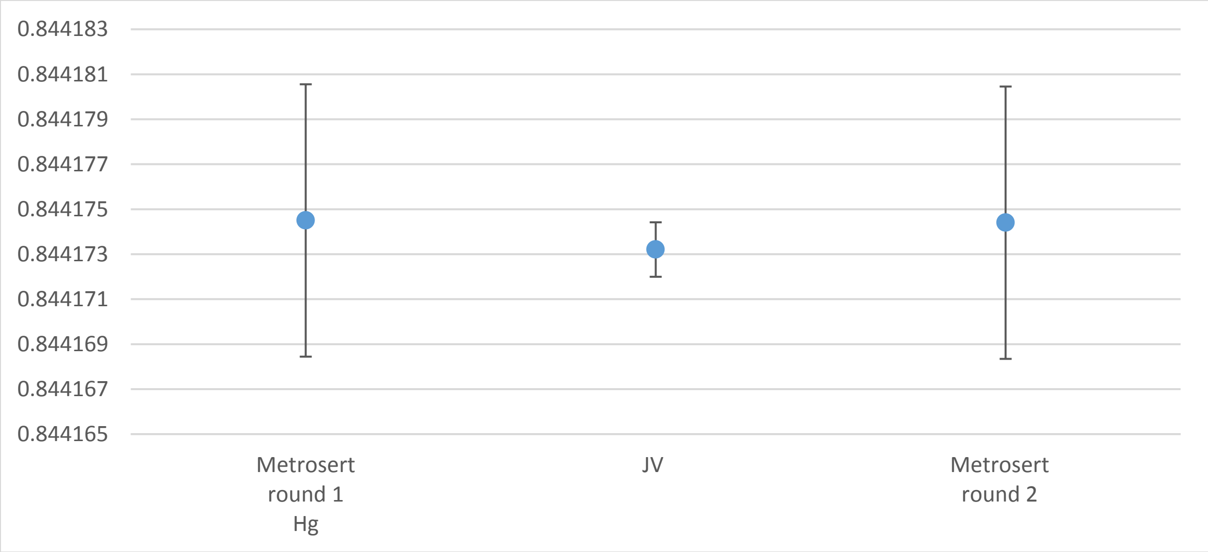


Figure 1: Reported W values at the triple point of Hg. Gradation on the y-axis corresponds to around 0,5 mK.

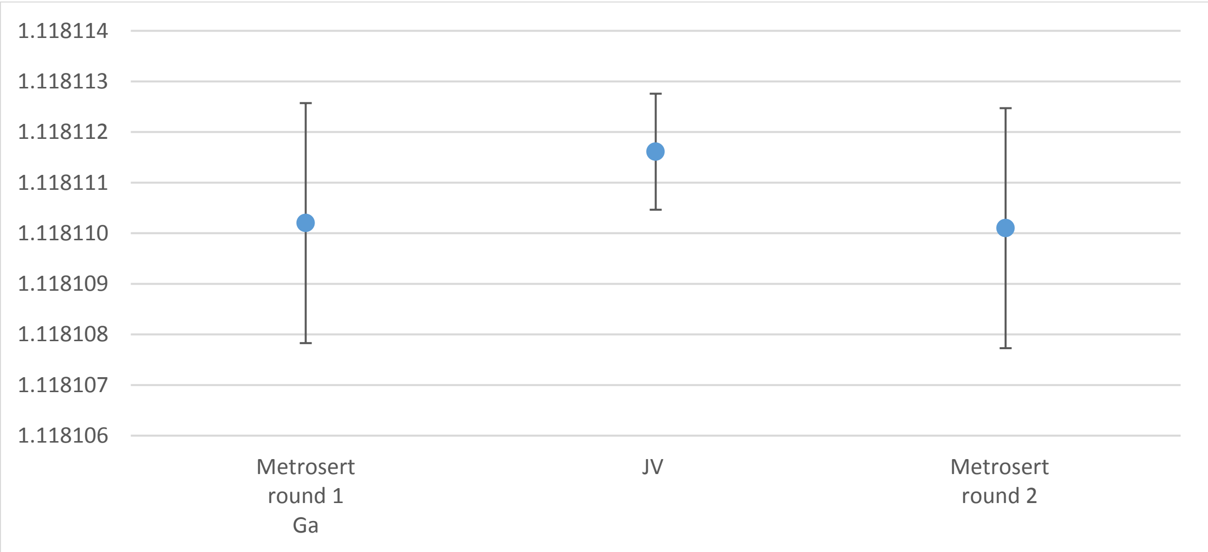


Figure 2: Reported W values at the melting point of Ga. The gradation on the y-axis corresponds to 0,25 mK.

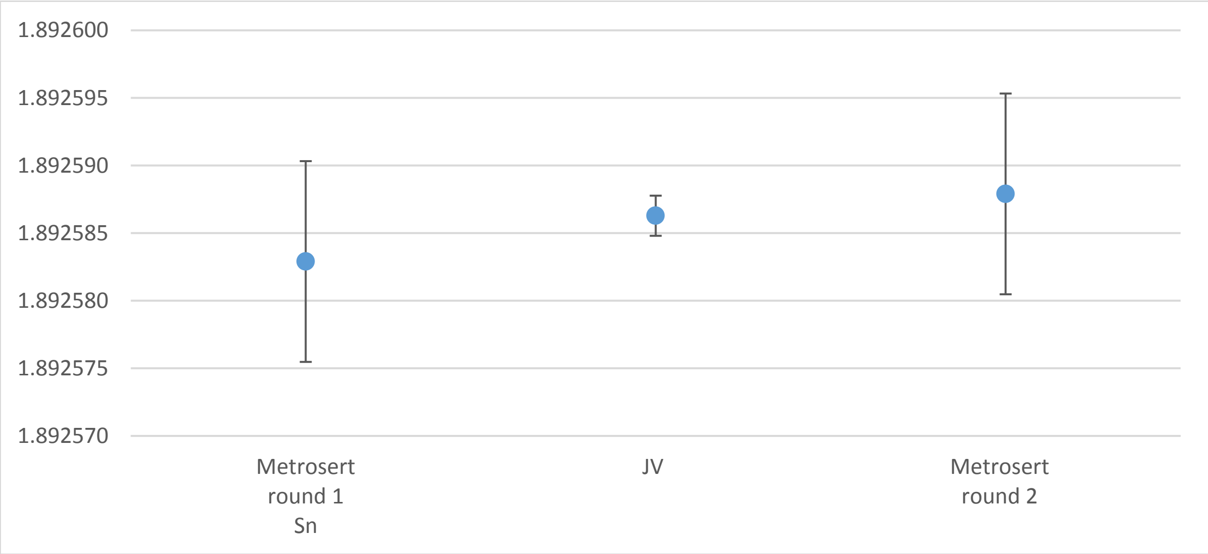


Figure 3: Reported W values at the freezing point of Sn. The gradation on the y-axis corresponds to 1,3 mK

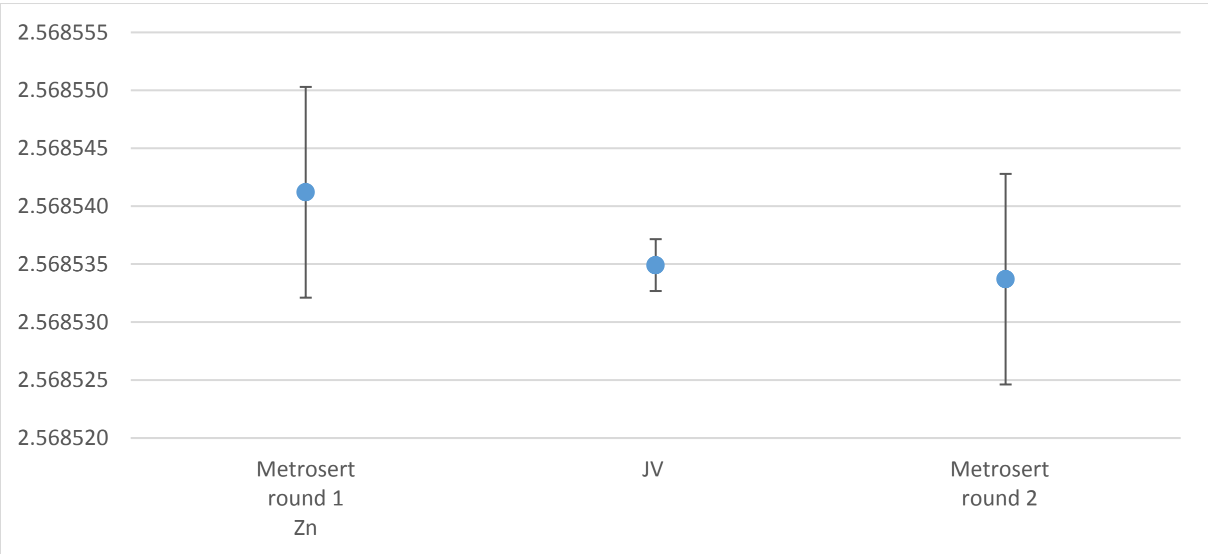


Figure 4: Reported W values at the freezing point of Zn. The gradation on the y-scale corresponds to 1,3 mK.

5.2 Triple point of water measurements

The reported triple point of water resistances are listed in Table 5. A column of W values is also shown in the table. The values were computed as a ratio of observed TPW resistance to the average TPW resistance across all the measurements. The purpose is just to be able to compute an equivalent temperature deviation from the ITS90 reference function; the procedure is explained in Section 6.1. The result of the temperature conversion is plotted in Section 6.4.

Table 5: Resistance at the triple point of water as reported by the participants. Values were recorded between all fixed point measurements. The W values are ratios between the observed resistance in each case divided by the average reported resistance, and were computed from the resistance values to enable a conversion to equivalent temperature change. See Section 6.4 for a plot of the data.

When		$\boldsymbol{R}$ (Ω)	$\boldsymbol{W}$ (using average $\boldsymbol{R}$)
Metrosert *(round 1)*			
	01 After annealing	25,3253917	1,0000033
	02 After Zn	25,3252580	0,9999981
	03 After Sn	25,3253510	1,0000017
	04 After Ga	25,3253573	1,0000020
	05 After Hg	25,3253036	0,9999999
Metrosert *(round 2)*			
	01 Before annealing	25,3253137	1,0000003
	03 After annealing	25,3252565	0,9999980
	04 After Zn	25,3252933	0,9999995
	05 After Sn	25,3252999	0,9999997
	06 After Ga	25,3253166	1,0000004
	07 After Hg	25,3252541	0,9999979
JV			
	01 Upon reception	25,3254651	1,0000062
	02 After annealing	25,3252512	0,9999978
	03 After Zn, 1	25,3252581	0,9999981
	04 After Zn, 2	25,3252507	0,9999978
	05 After Sn, 1	25,3253015	0,9999998
	06 After Sn, 2	25,3252985	0,9999997
	07 After Ga, 1	25,3252946	0,9999995
	08 After Ga, 2	25,3253054	0,9999999
	09 After Hg, 1	25,3253147	1,0000003
	10 After Hg, 2	25,3253138	1,0000003
Average		**25,3253071**	-

5.3 Immersion profiles

Immersion profiles were reported by the participants. Metrosert reported the data as W versus immersion, while JV reported resistance values as a function of immersion. The resistance values were converted to a W value with the aid of the reported W values at each fixed point, and then converted to a temperature using the ITS-90 fixed point temperatures and the procedure outlined in Section 6.1 to convert a small change in W to a small change in temperature. More details can be found in Appendix B along with the reported resistance values from JV.

Metrosert reported immersion profiles with reference to the tip of the thermometer rather than the midpoint of the sensing element. The reported positions have been shifted upwards by 3 cm to correct for this.

These transformations allow a presentation of the data using the same axis units. It should be noted that the immersion data were not subjected to the same corrections as the main data reported for the fixed points. Furthermore, the TPW value was not remeasured before and after the immersion profiles were recorded. The consequence is a small mismatch between the apparent temperature at full immersion and the actual fixed point temperature. However, the changes in observed resistance or W, when converted to an equivalent temperature change as a function of immersion, remain correct.

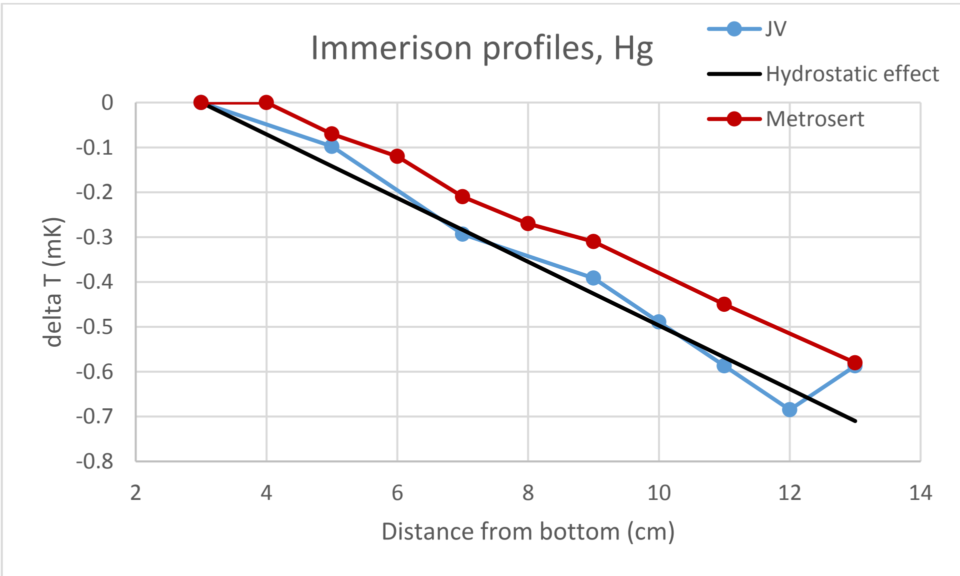


Figure 5 Immersion profiles at the triple point of Hg along with the ITS90 hydrostatic correction.

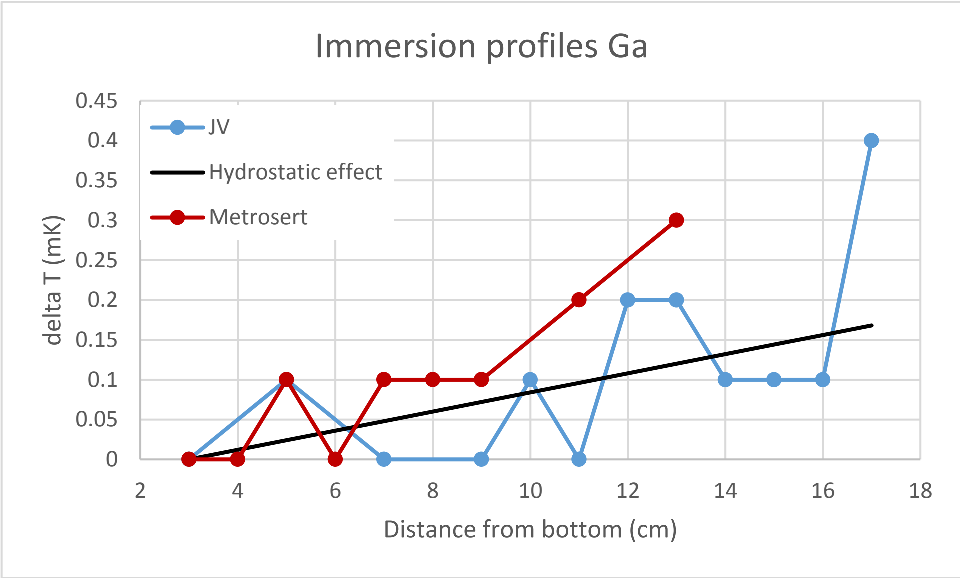
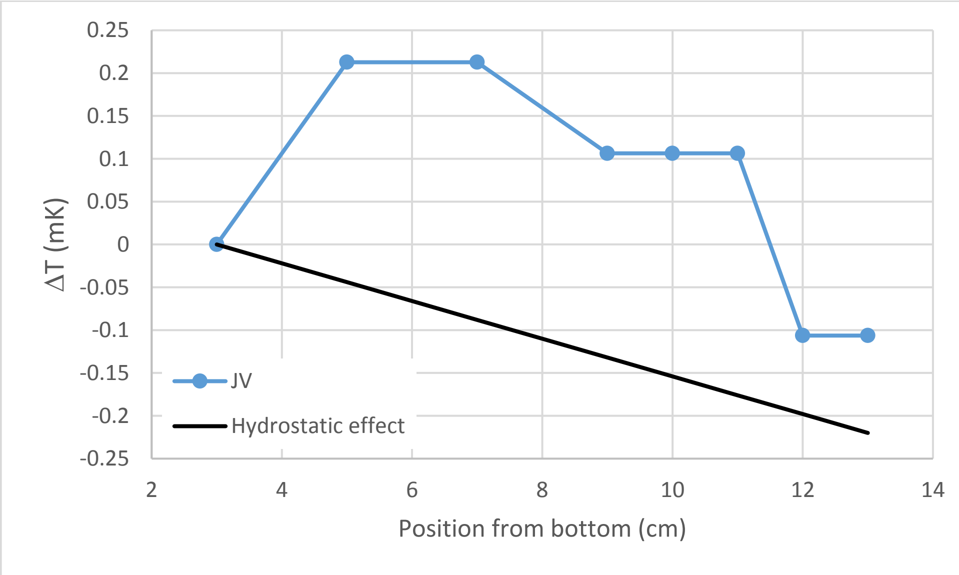


Figure 6 Immersion profiles at the melting point of Ga along with the ITS90 hydrostatic pressure correction.

The immersion profiles at the triple point of Hg and the melting point of Ga are shown in Figure 5 and Figure 6. The profiles are close to the expected hydrostatic correction from the ITS90 supplementary documents published by the BIPM (1).



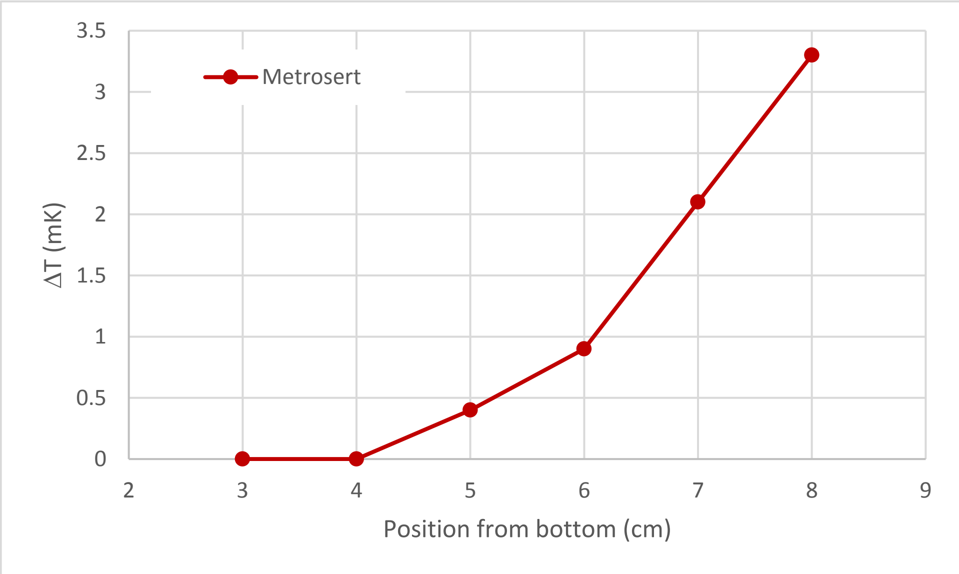


Figure 7 Immersion profiles at the freezing point of Sn. The measured profiles do not represent the hydrostatic pressure. At Metrosert the immersion is dominated by additional heating elements above the fixed point. At JV there seems to be a peak in temperature a little above the full immersion, which is likely to be caused by heat transfer.

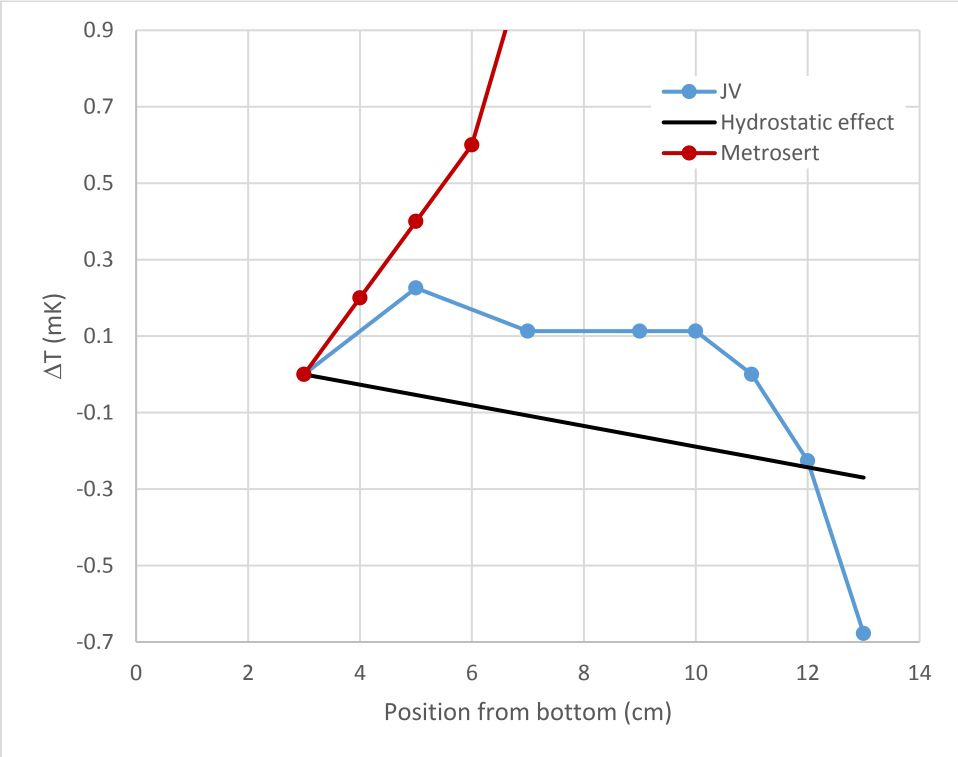


Figure 8 Immersion profiles at the freezing point of Zn. At Metrosert the immersion compensation heater dominates the profile. At JV the profile reaches a maximum some distance above the bottom. Heat transfer dominates the profiles rather than the hydrostatic pressure correction.

At the Sn and Zn points the immersion measured profiles are not determined by the hydrostatic pressure. Metrosert uses a furnace assembly with an additional heater element above the fixed point cell which is used to compensate for the immersion profile of typical thermometers. For the SPRT used in the comparison this leads to an increase in measured temperature as the sensing element is raised. At JV the fixed point cells are mounted in a heat pipe furnace liner with very uniform temperature along the axis. The behaviour is similar in both cases, with a small increase in temperature initially, before the temperature gradually falls again. The behaviour cannot only be attributed to changes in the transition temperature caused by the hydrostatic pressure of the molten metal. The details have not been investigated, but possible sources include an irregular freeze front inside the cell and heat transfer from the furnace through the thermometer stem, the walls in the fixed point enclosure, or heat loss through the bottom of the cell. An additional component of uncertainty due to this observation is included in the heat flux term in the uncertainty budget of JV.

5.4 Corrections to measured resistance

The participants have computed correction factors applied to the measured resistance. These corrections were converted to temperature using a method described in the protocol, and reported to the coordinator. The following tables list the values:

Table 6 Corrections applied by Metrosert.[*]*Uncertainties are standard uncertainties.*

	self-heating		Hydrostatic		pressure	
Before sending probe	ΔT (mK)	u (mK)	ΔT (mK)	u (mK)	ΔT (mK)	u (mK)
Zn	-1,90	0,05	-0,41	0,5	-	-
Sn	-1,64	0,05	-0,34	0,5	-	-
Ga	-1,31	0,05	0,24	0,2	-	-
Hg	-1,35	0,2	-1,31	0,5	-	-
After receiving probe						
Zn	-1,76	0,05	-0,41	0,5	-	-
Sn	-1,84	0,05	-0,34	0,5	-	-
Ga	-1,38	0,05	0,24	0,2	-	-
Hg	-1,40	0,2	-1,31	0,5	-	-

Table 7: Corrections applied by JV to the measured resistance at each fixed point. Uncertainties are standard uncertainties.

	self-heating		hydrostatic		pressure	
	ΔT (mK)	u (mK)	ΔT (mK)	u (mK)	ΔT (mK)	u (mK)
Zn	-2,07	0,12	-0,34	0,03	0	0,15
Sn	-1,93	0,11	-0,28	0,03	0	0,11
Ga	-1,46	0,09	0,16	0,01	0	0,07
Hg	-1,29	0,08	-1,10	0,08	0	0,18

6 Analysis of results

The reported data is used to compute a temperature difference between JV and Metrosert, an associated uncertainty, and equivalent temperature changes in the triple point of water measurements. Finally, the bilateral equivalence between JV and Metrosert is linked to the CCT-K9 via JV's linkage to that comparison.

6.1 Calculations

Temperature differences are computed from W differences by scaling with the derivative of the ITS-90 reference function for SPRTs:

$$\Delta W = \frac{dW_r}{dT}\Delta T \qquad (6\text{-}1)$$

[*] Note: values reported by Metrosert were errors rather than corrections. After communication between the pilot and the Metrosert representative the signs have been switched in this table.

ΔW is a small difference in the resistance ratio between two measurements, ΔT is the corresponding small difference in temperature, and W_r is the ITS-90 reference function for SPRTs. Equation 6-1 is the basis for all conversions between observed W and temperature.

The temperature difference between JV and Metrosert, $\Delta\theta$, is computed from the reported W values:

$$\Delta\theta = \frac{\overline{W_1} - W_2}{dW_r/dT} + C \qquad \text{6-2}$$

Since Metrosert measured twice, the W_1 value used in the equation is the average value of the results before and after measurements at JV. The term C accounts for changes in the measurement conditions, among them drift in the probe, and is assigned the value 0. However, it does add to the uncertainty.

The standard uncertainty in $\Delta\theta$, u_θ, is computed from the usual GUM method:

$$u_{\Delta\theta}^2 = u_{T,Metrosert}^2 + u_{T,JV}^2 + u_c^2 \qquad \text{6-3}$$

The uncertainty in C is assigned a rectangular distribution with width determined by the measurements at Metrosert (before and after measurements at JV), which implies a standard uncertainty u_c:

$$u_c = \frac{1}{\sqrt{12}} \frac{\left\| W_{1,after} - W_{1,before} \right\|}{dW_r/dT} \qquad \text{6-4}$$

In Equations 6-2 and 6-4 the conversion between ΔW and ΔT is shown explicitly.

For the triple point of water surveillance plot in Figure 9, resistances were converted to temperature deviation, τ, using Equation 6-1. The average of all reported resistances at the TPW was used as the reference resistance to compute W values, and the deviation from 1 was computed for each reported value:

$$\tau_i = \left(\frac{R_{TPW,i}}{R_{TPW}} - 1 \right) \frac{1}{dW_r/dT} \qquad \text{6-5}$$

6.2 Repeatability

The repeatability of the measurements at Metrosert are added to the uncertainty in the degree of equivalence according to Equation 6-4. This includes drift of the probe, but also the repeatability of equipment used at Metrosert. The triple point of water measurements are summarised in Section 6.4, and might indicate a small change in the probe. For the other fixed points the results are summarised in Table 8. The table shows

$$\left\| W_{1,after} - W_{1,before} \right\|$$

converted to temperature using Equation 6-1.

Table 8: Change in the observations at Metrosert before and after the probe was at JV. The table shows data for all the metal fixed points.

Fixed point	Change (mK)
Zn	2,15
Sn	-1,35
Ga	0,03
Hg	0,02

There is a noticeable change at the Sn and Zn points. The difference is large enough that the coordinator asked Metrosert to confirm the values. After examining their records the reported figures were found to be correct. The difference adds to the uncertainty in the degree of equivalence between Metrosert and JV at those fixed points.

6.3 Bilateral equivalence

The degree of equivalence between JV and Metrosert is summarised in Table 9. The calculation is explained in section 6.1.

Table 9: The degree of equivalence between JV and Metrosert. The table shows the temperature difference, the standard uncertainty, and expanded uncertainty.

	$\Delta\theta$ (mK)	u (mK)	$U_{95\%}$ (mK)
Hg	0,3	1,5	3,1
Ga	-0,4	0,7	1,3
Sn	-0,2	2,1	4,2
Zn	0,7	2,8	5,5

6.4 TPW surveillance

The reported resistances at the triple point of water are shown graphically in Figure 9, after conversion to an equivalent temperature deviation as described in Section 6.1.

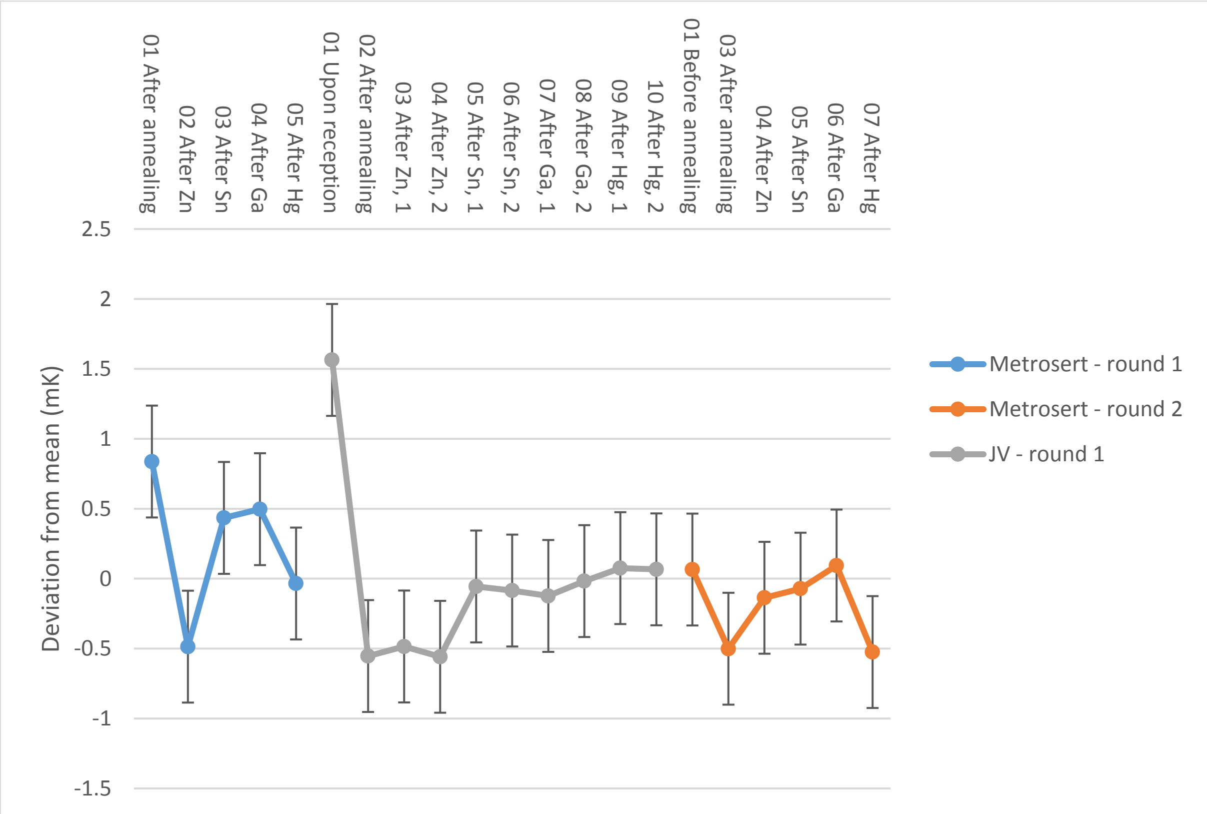


Figure 9 Deviation of TPW resistance values, from the average value of all TPW measurements (JV and Metrosert combined). The measured resistance is converted to temperature from the sensitivity of the reference function for SPRTs. The error bars are the reported standard uncertainty at the triple point of water from each participant.

6.5 Linkage to the CCT-K9 reference value

The linkage to CCT-K9 is accomplished via JV's values in EURAMET.T-K9:

$$\Delta T_{METROSERT} = \Delta\theta + \Delta T_{JV} \qquad 6\text{-}6$$

Here $\Delta T_{METROSERT}$ is the difference between Metrosert and the reference value of CCT-K9, while ΔT_{JV} is the difference between JV and the reference value. $\Delta\theta$ is defined above.

The uncertainty is computed from

$$u_{METROSERT}{}^2 = u_{\Delta\theta}{}^2 + u_{JV}{}^2 \qquad 6\text{-}7$$

Computing the actual values requires the results from EURAMET.T-K9, which are not yet available.

7 Discussion on the uncertainty calculations

The uncertainty budgets supplied by the participants suggest that the effective degrees of freedom is large for both participants, and the output distribution of the reported results is approximately normal. The temperature difference between JV and Metrosert has an additional term from the drift, which is modelled as a rectangular distribution. At the Zn and Sn points the drift has a noticeable contribution to the uncertainty, although its contribution remains smaller than both Justervesenet and Metroserts uncertainties in the measurements. The $\Delta\theta$ can still be regarded as approximately normal, as verified from a Monte Carlo computation of the temperature difference using the reported uncertainties as input distributions.

There could be some correlation between JV and Metrosert uncertainties. In particular, the hydrostatic head correction and the SPRT self-heating correction are computed in the same way. The hydrostatic head correction depends on the immersion depth in the cells and the density of the molten material. The latter must be very similar in order to comply with ITS-90 requirements. The immersion depth is measured by each participant separately, in different cells, and there is no reason to expect a strongly correlated error.

The SPRT self-heating is estimated from measurements at two different excitation currents, and a correction is computed by extrapolating the observations to zero current. Metrosert reports a noticeable difference before and after the measurements at JV, larger than the uncertainty in the correction. It is possible that Metrosert underestimates the uncertainty of the self-heating correction. However, the combined uncertainty reported by Metrosert is larger than JV's uncertainty at all points, and a closer inspection might reveal components to be estimated conservatively, and hence that an increase in the correction uncertainty would be balanced by a reduction in some other contributions.

8 Conclusion

JV and Metrosert have carried out a bilateral comparison with the objective to link Metrosert to the CCT-K9. The results reported by JV and Metrosert are in agreement. The circulating instrument, an SPRT, is susceptible to drift during transportation. Drift in the probe leads to an increased uncertainty at Sn and Zn points.

9 Reference

1. **Pearce, J V, et al.** *Guide to the Realization of the ITS-90: Metal Fixed Points for Contact Thermometry.* Paris : BIPM, 2018.

2. *The International Temperature Scale of 1990 (ITS-90).* **Preston-Thomas, H.** 1990, Metrologia, Vol. 27, ss. 3-10.

Appendix A. Uncertainty budgets

Justervesenet

	TPW		Hg		Ga		Sn		Zn		
	mK	**Df**	**mK**	**df**	**mK**	**df**	**mK**	**df**	**mK**	**df**	**Type A or B (*)**
Phase transition realization repeatability	0.02	1	0.04	1	0.17	1	0.003	1	0.11	1	A
Repeatability of readings (FP)	0.03	59	0.03	59	0.03	59	0.03	59	0.03	59	A
Bridge (repeatability, non-linearity, AC	0.06	∞	0.06	∞	0.06	∞	0.06	∞	0.07	∞	B
Reference resistor stability	0.00	∞	0.00	∞	0.00	∞	0.01	∞	0.01	∞	B
Chemical impurities	0.1	∞	0.11	∞	0.01	∞	0.17	∞	0.33	∞	B
Hydrostatic-head	0.01	∞	0.08	∞	0.01	∞	0.03	∞	0.03	∞	B
Repeatability of readings (TPW)			0.02	59	0.03	59	0.05	59	0.08	59	A
Propagated TPW			0.15	∞	0.18	∞	0.29	∞	0.45	∞	B
SPRT self-heating	0.08	∞	0.08	∞	0.09	∞	0.11	∞	0.12	∞	B
Heat flux	0.03	∞	0.06	∞	0.03	∞	0.09	∞	0.16	∞	B
Insulation leakage	0.00	∞	0.00	∞	0.00	∞	0.00	∞	0.00	∞	B
SPRT Pt oxidation	-	-	-	-	-	-	-	-	-	-	
Gas pressure	0.00	∞	0.18	∞	0.07	∞	0.11	∞	0.15	∞	B
Combined standard uncertainty	0.15		0.30		0.29		0.40		0.64		
Expanded uncertainty (95% coverage, using effective df)	0.30		0.60		0.57		0.79		1.3		

Metrosert

	TPW		Hg		Ga		Sn		Zn		
	mK	**Df**	**mK**	**df**	**mK**	**Df**	**mK**	**df**	**mK**	**df**	**Type A or B (*)**
Uncertainty of fixed point(**)	0,08	49	1,00	49	0,40	49	1,91	49	2,48	49	A
Bridge (repeatability, non-linearity, AC quadrature)	0,02	99	0,02	99	0,02	99	0,03	99	0,04	99	A
Reference resistor stability	0,02	48	0,02	48	0,02	48	0,02	48	0,02	48	B
Hydrostatic-head	0,20	195	0,50	195	0,20	195	0,50	195	0,50	195	B
Propagated TPW			0,32	4	0,32	4	0,32	4	0,32	4	B
SPRT self-heating	0,05	97	0,20	97	0,05	97	0,05	97	0,05	97	B
Insulation leakage	0,20	999	0,20	999	0,20	99	0,20	999	0,20	999	B
SPRT Pt Oxidation	0,10	199	0,10	199	0,10	199	0,10	199	0,10	199	B
Gas pressure	-	-	-	-	-	-	-	-	-	-	-
Combined Standard Uncertainty	0,3	935	1,2	89	0,6	41	2,0	59	2,6	55	
Expanded Uncertainty (95% coverage, using effective df)	1		3		2		4		6		

(*) write A or B depending on the method used
df: degree of freedom

(**)includes: Phase Transition Realization Repeatability, Chemical Impurities and Heat Flux.

Appendix B. Reported immersion profiles from JV and conversion to temperature

The resistance values as a function of immersion depth reported by JV are shown in Table 10.

Table 10 Reported resistance values from JV as a function of immersion depth.

Position (cm)	Zn ($R_{0\ mA}$, Ω)	Sn ($R_{0\ mA}$, Ω)	Ga ($R_{0\ mA}$, Ω)	Hg ($R_{0\ mA}$, Ω)
3	65.04884	47.93034	28.31649	21.37907
5	65.04886	47.93036	28.3165	21.37906
7	65.04885	47.93036	28.31649	21.37904
9	65.04885	47.93035	28.31649	21.37903
10	65.04885	47.93035	28.3165	21.37902
11	65.04884	47.93035	28.31649	21.37901
12	65.04882	47.93033	28.31651	21.37900
13	65.04878	47.93033	28.31651	21.37901
14	65.04872	47.93024	28.3165	21.37900
15	65.04847	47.93008	28.3165	21.37904
16	65.04785	47.92971	28.3165	21.37919
17	65.04632	47.92913	28.31653	21.37996
18	65.04296	47.92842	28.31652	21.38140

The resistance values were converted to a W value from

$$W(x) = \frac{R(x=0)}{R(x)} W_{JV}$$

where $R(x)$ is the reported resistance at immersion depth x, and W_{JV} is JV's reported W value at the relevant fixed point. The W values are used to compute a small change in W with immersion, and this is converted to a temperature offset as a function of immersion using Equation 6-1:

$$T(x) = T_{90} + \frac{W(x) - W(x=0)}{dW_r/dT}$$

Appendix C. Document history

27.09.2019	*Modifications in response to review* Immersion profiles recomputed for JV, and graphs updated. No substantial changes. Modified section 6.2 slightly, to make it clear(?) that it does not imply a further term in Eqn 6-2. Layout changes to avoid tables spanning more than one page.
23.01.2019	*Modifications in response to reviewer comments* Immersion profiles were recomputed and new graphs drawn. Comments to immersion profiles changed. Tables given table numbers throughout. Description of "drift" modified, but substantially retained (still used in DoE etc). Number of digits in table 6 changed. Added information on TPW cells and resolution/linearity of bridges.
05.06.2018	*Modifications in response to reviewer comments:* Changed the number decimal digits in tables Added sentence in equipment descriptions (section 4) Added uncertainty in W in Table 4 Fixed error in drift values in Table 4 Removed En-value JV has reduced the uncertainty due to chemical impurities and self-heating Modified axis ranges in immersion profile figures. Added a paragraph to explain odd behaviour of Metrosert immersion profiles. Added a brief discussion for Metrosert offset in the immersion profiles, particularly for Ga.
08.03.2018	Bilateral draft A, version 2, completed. Fixed bad references. Added authors. Moved protocol to the final appendix. Corrected some language and grammar errors. Added a description of the environmental conditions and control at Metrosert (section 4).
05.02.2018	Bilateral Draft A, version 1, completed. Sent to participants for comments.
17.01.2018	Following a request from the pilot, Metrosert confirmed that transcribed W values at Sn and Zn are correct.

Appendix D. Approved protocol

This copy of the approved protocol leaves out the appendices, but a table indicates where the contents of those appendices can be found in this report.

EURAMET.T-K9.1 final protocol

Bilateral comparison

ITS-90 SPRT Calibration from the Hg TP to the Zn FP

1 Objective

This comparison is a bilateral comparison between Justervesenet (Norway, abbreviated JV) and METROSERT (Estonia) with the purpose of providing the latter with linkage to the key comparison CCT-K9. Justervesenet currently takes part in the regional comparison EURAMET.T-K9, which links to the CCT-K9. Justervesenets linking laboratory is LNE-Cnam.

The range of temperature covered in this comparison is from the triple point of Hg (234.3156 K) to the freezing point of Zn (692.677 K). The transfer standard used will be a long-stem SPRT.

This protocol matches closely the corresponding EURAMET.T-K9 regional comparison protocol. An important exception is in the fixed points used, which excludes the triple point of Ar (83.8058 K) and the freezing point of In (429.7485 K) because Metrosert does not currently realise those fixed points.

2 Topology of the comparison

The comparison is bilateral with JV as pilot and METROSERT as the only participant.

3 Participants

JV, Norway. Pilot.

METROSERT, Estonia, participant.

4 Schedule

4.1 Timeline

Protocol Agreement	May, 2017
Measurements at METROSERT	July, 2017
Transfer to JV and measurements at JV	August, 2017
Return to METROSERT for repeat measurements	September, 2017
Draft A Report Completed	January, 2018

4.2 Dependencies

The data analysis and reporting depends on the EURAMET.T-K9 report. JV's linkage and associated uncertainty is needed to compute the corresponding values for METROSERT.

5 Circulating instrument

Long stem SPRT

Model:	670 SQ Pt25
Serial no:	052
Manufacturer:	Isotech
Owner:	Metrosert

The device must be handled with care. Only qualified laboratory personnel should handle it. The circulating instrument will be hand carried between the laboratories. Metrosert is responsible for the transport from Estonia to Norway, and JV is responsible for returning the instrument to Metrosert.

6 Measurements

The resistance at the triple point of water (TPW) shall be measured before the measurements are started, after the measurements are completed, and also followed up according to each laboratory's current calibration procedure.

The SPRT W-value is measured at each of the following fixed points:

- The triple point of Hg
- The melting point of Ga
- The freezing point of Sn
- The freezing point of Zn

The measurements are carried out in sequence from highest to lowest temperature.

The cell immersion depth profile is measured once for each cell.

7 Reporting

7.1 Main results

Metrosert reports the results from the first round of measurements as soon as they are available.

Reporting is performed using the reporting worksheet in Appendix A.

The results are given in terms of W, the ratio of the electrical resistance at each of the fixed points to the electrical resistance at the TPW. The uncertainty is the standard uncertainty along with degrees of freedom, converted to mK. If more than one repetition is performed, the reported result is the average value.

The calibration results are supplied with all corrections applied such that the W values are equivalent to the ITS-90 assigned temperature values for 0 mA.

All measured TPW resistance values shall be reported in a separate table. The table will be used to judge changes in the SPRT probe as the calibration progresses.

7.2 Auxiliary data

The auxiliary data is not used in the analysis explained in section 10, but is inspected in the event of spurious results.

- **The measurement equation** used to compute each calibration result shall be supplied, along with an indication of which inputs vary randomly for each realized equilibrium and which inputs are systematic across all equilibriums for each fixed point within this comparison
- **Heat Flux (Immersion) profile** for each fixed-point cell using the SPRT of this comparison: [R, 0 mA] and corresponding [immersion depth (sensor midpoint), cm].
- **The corrections applied to the measured $\boldsymbol{R}$** at each fixed point, see Appendix B.

8 Responsibilities

8.1 JV

- To keep track of the progress and report to the CCT.
- To check the initial value of R_{TPW} of the SPRT upon arrival at JVs premises, before and after annealing, against the final value reported by METROSERT. In the case of significant discrepancy, JV and METROSERT will discuss how to proceed, and if necessary consult the EURAMET TCT.
- To carry out the calibration of the SPRTs using the same equipment as was used in EURAMET.T-K9, or equipment which is known to be compatible with that equipment.

- To prepare tables of reported results and uncertainties, and for each fixed point to calculate the differences $\Delta T_{METROSERT}$ and associated uncertainties $u^2_{METROSERT}$ (see section 10)
- To hand carry the instrument to the Metrosert premises after measurements at JV are completed

8.2 Metrosert

- To report any issues that may cause a delay to JV as soon as possible
- To hand carry the instrument to JV's premises after the first round of measurements are completed

9 Miscellaneous

- **Instructions for reporting the results:** The templates in Appendices A, B and C should be used for reporting the results.
- **Timetable for communicating the results:** METROSERT must report the results of their measurements to JV at the time the SPRT is transferred to JV. METROSERT must notify JV if the results of the recalibration (measurements performed after the return of the SPRT) deviates from the initial measurements by more than the expanded uncertainty in the measurements at METROSERT. The participants will jointly work to reveal the reason for the discrepancy, and if necessary consult the EURAMET TCT for advice.
- **Financial aspects of the comparison:** JV and METROSERT are responsible for their own costs associated with the measurements (labour, equipment, repairs etc), as well as the transportation of the circulating instrument between the laboratories (see section 5).
- **Instrument damage:** in the event of damage to the circulating instrument, the participant who is presently in possession of the device will bear the cost of a replacement unit.

10 Method of Analysis and link to the CCT-K9 reference value.

10.1 Notations

ΔT_{JV}	The linkage value for JV in EURAMET.T-K9
$T_{METROSERT}$	The temperature measured by METROSERT, converted from reported W value
T_{JV}	The temperature measured by JV, converted from reported W value.
C	Correction term associated with transport and handling of the SPRT
$u_{\Delta T,JV}$	Uncertainty in JV's linkage to EURAMET.T-K9
$u_{T,METROSERT}$	Uncertainty of the measurements at METROSERT
$u_{T,JV}$	Uncertainty of the measurements at JV in EURAMET.T-K9.1
u_C	Uncertainty associated with handling and transport

Other symbols used follow standard notation. For easier notation we do not indicate the fixed point; each quantity is assigned numbers for each fixed point used in the comparison.

10.2 Computations

The reference value is the Key Comparison Reference Value of CCT-K9.

Linkage is provided by

$$\Delta T_{METROSERT} = \left(T_{METROSERT} - T_{JV}\right) + \Delta T_{JV}$$

$\Delta T_{JV} = T_{JV,EURAMET.T-K9} - T_{KCRV}$ and the associated $u_{\Delta T,JV}$ will be available from the final report of the EURAMET.T-K9, linked via JV's pilot in that comparison (LNE-Cnam).

The temperature difference between JV and METROSERT is computed from

$$T_{METROSERT} - T_{JV} = \frac{W_{METROSERT} - W_{JV}}{dW_r/dT} + C$$

The ITS-90 reference function is used to compute the sensitivity at each fixed point, which is the same as done in the EURAMET.T-K9 regional comparison. The value of $W_{METROSERT}$ is the average of the measurements before and after measurements at JV. The additional term C represents a correction associated with handling and transport. It is assigned an expected value of 0, but with an uncertainty given by a rectangular distribution with METROSERT measurements before and after the measurements at JV as the width:

$$u_C = \frac{\left|W_{METROSERT,1} - W_{METROSERT,2}\right|}{\sqrt{12} \cdot dW_r/dT}$$

The uncertainty of $\Delta T_{METROSERT}$, $u_{METROSERT}$, is computed from

$$u_{METROSERT}^2 = u_c^2 + u_{\Delta T,JV}^2 + u_{T,METROSERT}^2 + u_{T,JV}^2$$

A preliminary report will be issued which presents results from the bilateral comparison, in the event that the bilateral comparison is finished before the draft A report from the regional comparison is available. This preliminary report is considered confidential and may not be disclosed to third parties unless both participants agree. The final report, including the linkage to EURAMET.T-K9, will be written as soon as the results from that comparison are ready.

The following appendices from the protocol are not included here:

Appendix A	**Measurement reporting sheet** See reported values in Section 5
Appendix B	**Corrections applied to the measured values** See reported values in Section 5.4
Appendix C	**Suggested uncertainty budget** See reported budgets in Appendix A
Appendix D	**Contact details of the participants** See section 2