Report to the CCT on Bilateral Key Comparison EURAMET.T-K7.1

EURAMET Project 1082

Bilateral key comparison of the water triple point cells

EURAMET.T-K7.1

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FINAL REPORT

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1 Organization of the comparison

1.1 Introduction

Key comparison of water triple point cells EURAMET.T-K7.1 (EURAMET Project No. 1082) was organized as additional comparison to previously realized regional comparison of water triple point cells EURAMET.T-K7 (EURAMET. Project No. 899). The decision to organize this comparison was taken during the EURAMET Thermometry Technical Committee meeting that was held in Delft from 31 March to 2 April 2008. The reason of organizing this comparison was non-satisfactory result of some participants during realization of EURAMET.T-K7 resulting from instability of the TPW cell used as a transfer cell by these laboratories.

EURAMET.T-K7.1 was originally organized as key comparison with two participants, GUM and CMI. Due to the faulty of the transfer TPW cell, GUM was withdrawn from the comparison and it was transformed into the bilateral key comparison between the Slovak Institute of Metrology (SMU) and the Czech Metrology Institute (CMI). The pattern of this comparison is very close to EUROMET.T-K7.

1.2 Participating laboratories

Participating laboratories with corresponding contact persons are listed in the following table.

Acronym	Name of Institute	Country	Contact person
		Pilot	
SMU	Slovak Institute of Metrology	Slovak Republic	Stanislav Ďuriš, +421 2 602 94 299, duris@smu.gov.sk
		Participant	
CMI	Czech Metrology Institute	Czech Republic	Radek Strnad, +420 266 020 121, rstrnad@cmi.cz

Table 1: List of participating laboratories with corresponding contact persons at the time of comparisons.

1.3 Comparison method

Comparison was evaluated in such a way to provide the link to the EUROMET.T-K7. The links were established for the CMI national reference and for the new actual SMU national reference.

The comparison was conducted in the following steps:

1. CMI selected one transfer cell for the comparison and compared it with CMI national reference (group of cells).
2. SMU selected one transfer cell (cell which was used as transfer cell during EUROMET.T-K7) for the comparison and compared it with SMU national reference.
3. SMU compared the previous national reference cell (national reference cell during and EUROMET.T-K7) with the actual national reference SMU.
4. CMI transported its transfer cell to the SMU. SMU compared transfer cells (SMU and CMI).

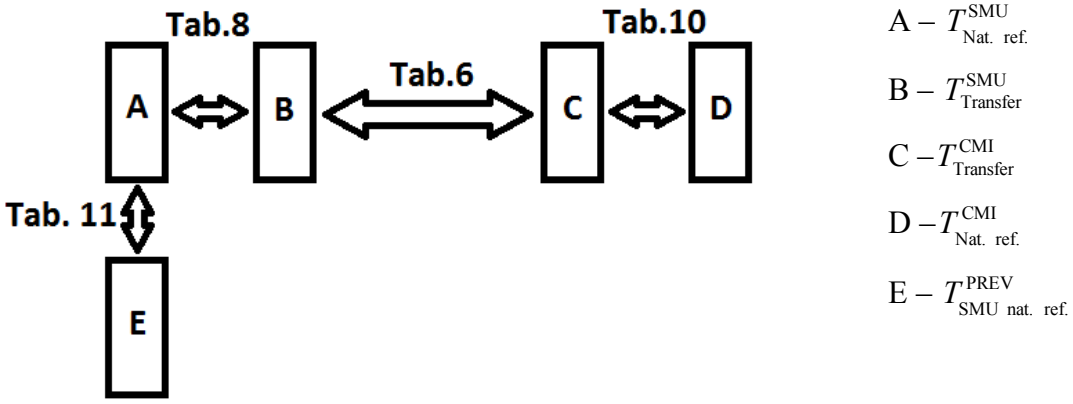

Fig. 1: Comparison schema. Transfer cells were compared with national references (Table 8 and Table 10). Thereafter transfer cells af SMU and CMI were compared (Table 6). Because of replacement of SMU national reference after completing of EUROMET.T-K7, there was establish its link by comparison of actual SMU national reference cell with previous SMU national reference cell (Table 11).

1.4 SMU transfer cell, CMI transfer cells

As a transfer cell for the comparison was selected the TPW cell SMU-1 that was used as transfer cell during CCT-K7 and also during EUROMET.T-K7. Overview of information about this cell is presented in Table 2.

	Reference cell for the comparison
Cell designation	SMU-1
Manufacturer or type	VNIIM
Model of cell	
Year of fabrication or purchase	2000
Accessories or comments on special use	-
Inner diameter of well / mm	11
Cell diameter / mm	50
Depth of well below water surface / mm	266

Table 2: Overview of information on the transfer cell for the comparison.

CMI selected the transfer cell. Following table provides the overview of information available on the CMI transfer cell.

Cell designation	**CMI E11-228**
Manufacturer or type	Isotech
Model of cell	Jarret
Year of fabrication or purchase	2002
Accessories or comments on special use	-
Inner diameter of well / mm	11
Cell diameter / mm	50
Depth of well below water surface / mm	290

Table 3: Overview of information available on the transfer cell selected by the CMI.

2 Equipment, techniques and uncertainties

2.1 Laboratory equipment and techniques

The equipment, the techniques and the measuring conditions at participating laboratories are summarized in Table 4. Regarding the technique for the preparation of the ice mantle, participating laboratories used the procedures that normally apply (see last row of Table 4).

	SMU	**CMI**
Resistance ratio bridge	ΑΣΛ F900, AC	ΑΣΛ F900, AC
Measurement current (frequency)	1 mA (75 Hz)	1 mA, (75 Hz)
Number of repeated measurements	24	26
Frequency of repeated measurements	0.1 Hz	0,03 Hz
Reference resistor	100 Ω, Tinsley 5686 A	25 Ω, Tinsley 5685 A
Temp. control of reference resistor	± 1 mK	±10 mK
SPRT and length of the sensor	Isotech 670, 40 mm	Fluke, HART Scientific ,30 mm
Storage container for WTP cells	ISOTECH type ITL-M-18233	ISOTECH, type ITL-M-18233
Technique ice mantle preparation	LN-cooled bar	In a cryostat filled with ethanol undercooled to approximately –35 °C. The ethanol is introduced into the system by silicon hoses through a double bored rubber plug at the bottom of the TPW and runs out through another plug app. 10 mm below the surface of the water in the TPW cell.

Table 4: Overview of the equipment and measuring conditions in the participating laboratories.

2.2 Uncertainties

The uncertainty budgets submitted by the participating laboratories are reported in the Table 5.

Origin	$T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}}$; $T^{SMU}_{\mathrm{Transfer}}$	$T^{\mathrm{CMI}}_{\mathrm{Nat.ref.}}$; $T^{\mathrm{CMI}}_{\mathrm{Transfer}}$	$T^{\mathrm{SMU}}_{\mathrm{Transfer}}$; $T^{\mathrm{CMI}}_{\mathrm{Transfer}}$	$T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}}$; $T^{\mathrm{PREV}}_{\mathrm{SMU\ Nat.\ ref.}}$
Components related to the comparison of a pair of cell (transfer cell to national reference)				
Repeatability for a single ice mantle (including bridge noise)	10	20	12	8
Reproducibility for different ice mantles	15	40	20	12
Reproducibility for different types of SPRTs		8		7
Hydrostatic head of transfer cell	3	10	5	3
Hydrostatic head of reference cell	3	10	0	3
SPRT self-heating (from both cells)	5	5	7	5
Perturbing heat exchanges	12	20	15	12
Total uncertainty (k=1) for the comparison of a pair of cells	**23**	**51**	**29**	**21**
National reference (components related only to properties of the reference cell(s))				
Chemical impurities	20	20		20
Isotopic composition	3	7		3
Residual gas pressure in the cell	5	10		5
Reproducibility	30	20		30
Total uncertainty (k=1) for the comparison of the transfer cell to the national reference	**43**	**60**	**29**	**42**

Table 5: Uncertainty budgets of the participating laboratories. All components are stated in μK and $k = 1$.

2.3 Comparison of CMI transfer cell and SMU transfer cell

CMI transfer cell was directly compared with the reference cell for the comparison. Measurements were performed on three separately prepared ice mantles. Ice mantles were prepared using the standard SMU technique (bar cooled by liquid nitrogen was inserted into the thermometer well filled by alcohol) on the same day. The measurement did not start earlier than 7 days after the preparation of ice mantles.

Arithmetic mean was used to combine the results for different ice mantles into single value. The results of the CMI transfer cell measurements are presented in Table 6.

$T_{\mathrm{Transfer}}^{\mathrm{SMU}} - T_{\mathrm{Transfer}}^{\mathrm{CMI}}$ / µK			
Measurenemt No.:	**First ice mantle**	**Second ice mantle**	**Third ice mantle**
1	-10.8	-13.7	-20.1
2	-30.2	-10.8	0.3
3	-12.7	-17.0	-17.5
4	-14.9	-7.1	-20.1
5	-25.4	-9.2	2.1
6	-28.7	-8.1	-9.9
7	4.9	11.2	0.2
8	6.1	3.7	-13.1
9	12.9	10.2	0.3
10	7.4	6.1	2.1
11		1.9	-14.9
Mean	**-6.7 µK**		
$u(T_{\mathrm{Transfer}}^{\mathrm{SMU}} - T_{\mathrm{Transfer}}^{\mathrm{CMI}})$	**29 µK**		

Table 6: Temperature differences between SMU transfer cell and CMI transfer cell with associated combined standard uncertainty ($k = 1$).

2.4 Comparison of actual SMU national reference with SMU transfer cell

After completing EUROMET.T-K7 the SMU replaced national reference cell by the cell with known isotopic composition. Information about the actual SMU TPW national reference is presented in Table 7.

Temperature difference between SMU transfer cell and actual SMU national reference calculated as the mean of the results for two series of measurements (each series was performed on separately prepared ice mantles) is $T_{\text{Transfer}}^{\text{SMU}} - T_{\text{Nat. ref.}}^{SMU}$ = -81.4 µK. Combined standard uncertainty, is estimated as 43 µK.

Definition of national reference	One cell.
Reference cell	Hart Scientific 5901-DQ, s.n.: DQ-1019
Correction of isotopic composition applied?	Yes, 1.5 µK.
Correction of impurities effect applied?	No, effect of impurities is included in uncertainty.

Table 7: Overview of information on the definition of the SMU national reference.

Meas. No.:	1. ice mantle	2. ice mantle
	$T_{\text{Transfer}}^{\text{SMU}} - T_{\text{Nat. ref.}}^{\text{SMU}}$ / µK	$T_{\text{Transfer}}^{\text{SMU}} - T_{\text{Nat. ref.}}^{\text{SMU}}$ / µK
1	-70.6	-90.9
2	-119.7	-90.1
3	-128.6	-69.9
4	-48.9	-105.6
5	-97.2	-78.1
6	-82.3	-86.3
7	-57.5	-107.9
8	-56.7	-53.9
9	-73.6	-61.6
10	-78.5	-70.5
Mean	**-81.4 µK**	
$u(T_{\text{ref.}} - T_{\text{SMU nat. ref.}})$	**43 µK**	

Table 8: Temperature differences between SMU transfer cell for the comparison and actual SMU national reference with associated combined standard uncertainty ($k = 1$). Uncertainty includes the contribution for deviation from the ideal water triple point realization.

2.5 Comparison of CMI transfer cell with CMI national reference

CMI determined the temperature difference between its transfer cell and CMI national reference together with associated combined standard uncertainty. CMI made measurements on two separately prepared ice mantles. Whereas national reference of CMI consists of three cells, difference between national reference and CMI transfer cell was stated as average value of differences of each national standard cell and CMI transfer cell.

Results of measurements performed by the CMI are presented in Table 10.Information about the definition of national reference provided by CMI is summarized in Table 9.

	CMI
Definition of national reference	Group of three cells
Reference cell	Three cells: Hart Scientific 5901D-G1102 Isotech B11-50Q-640 Isotech A11-50-723Q
Correction of isotopic composition applied?	Hart Scientific 5901D-G1102: included in the uncertainty, ±7 µK SMOW Isotech B11-50Q-640: yes, 13 ±3 µK Isotech A11-50-723Q: yes, 16 ±3 µK
Correction of impurities effect applied?	no

Table 9: Overview information available from the CMI on the definition of the national references.

Measurement. No.:	**1. ice mantle**	**2. ice mantle**
	$T_{\text{Transfer}}^{\text{CMI}} - T_{\text{Nat. ref.}}^{\text{CMI}}$ / **µK**	$T_{\text{Transfer}}^{\text{CMI}} - T_{\text{Nat. ref.}}^{\text{CMI}}$ / **µK**
1	-26.8	-71.5
2	-61.0	-66.7
3	-57.5	-76.5
4	-55.4	-55.3
5	-45.9	-76.2
6	-88.3	-81.3
7	-97.0	-87.5
8	-90.3	-102.2
9	-92.8	-104.0
10	-89.6	-115.6
11		-115.8
Mean	**-78.9 µK**	
$u(T_{\text{CMI transfer}} - T_{\text{CMI nat. ref.}})$	**60 µK**	

Table 10: Temperature differences between CMI transfer cell and CMI national reference with associated combined standard uncertainty ($k = 1$).

3 Comparison of actual SMU national reference and previous SMU national reference

Comparison of actual SMU national reference cell ($T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}}$) and previous SMU national reference ($T^{\mathrm{PREV}}_{\mathrm{SMU\ Nat.\ ref.}}$) is presented in the following table 11. Previous SMU national reference is linked to EUROMET.T-K7.

Meas. No.:	**1. ice mantle**	**2. ice mantle**
	$T^{SMU}_{\mathrm{Nat.Ref}} - T^{\mathrm{PREV}}_{\mathrm{SMU\ Nat.\ ref.}}$ **/ µK**	$T^{SMU}_{\mathrm{Nat.Ref}} - T^{\mathrm{PREV}}_{\mathrm{SMU\ Nat.\ ref.}}$ **/ µK**
1	1.6	-30.3
2	8.1	24.2
3	-12.2	1.2
4	-10.6	-31.6
5	136.4	23.3
6	-92.1	5.1
7	31.3	78.5
8	-61.9	53.9
9	-33.9	78.1
10	-75.6	37.4
Mean	**6.5 µK**	
$\boldsymbol{u}(T^{SMU}_{\mathrm{Nat.Ref}} - T^{\mathrm{PREV}}_{\mathrm{SMU\ Nat.\ ref.}})$	**42 µK**	

Table 11: Comparison of SMU national reference and previous SMU national refrerence in order to get the link to EUROMET.T-K7 with associated combined standard uncertainty ($k = 1$).

$T^{SMU}_{\mathrm{Nat.Ref}} - T^{\mathrm{ERV}}_{\mathrm{SMU\ Nat.\ ref.}}$ **/ µK**	**-12,5**
$\boldsymbol{u}(T^{SMU}_{\mathrm{Nat.Ref}} - T^{\mathrm{ERV}}_{\mathrm{SMU\ Nat.\ ref.}})$ **/ µK**	**63**

Table 12: Linkeage of actual SMU national reference cell ($T^{SMU}_{\mathrm{Nat.Ref}}$) to EUROMET.T-K7 with associated combined standard uncertainty ($k = 1$).

4 Pair Equivalence

Bilateral equivalence between SMU and CMI is expressed by the temperature difference between their national references and its uncertainty. The temperature difference $T^{\mathrm{CMI}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{CMI}}_{\mathrm{Transfer}}$ as calculated from the individual differences from:

$$T^{\mathrm{CMI}}_{\mathrm{Nat.\,ref.}} - T^{\mathrm{SMU}}_{\mathrm{Nat.\,ref.}} = \left(T^{\mathrm{CMI}}_{\mathrm{Nat.\,ref.}} - T^{\mathrm{CMI}}_{\mathrm{Transfer}}\right) + \left(T^{\mathrm{CMI}}_{\mathrm{Transfer}} - T^{\mathrm{SMU}}_{\mathrm{Transfer}}\right) + \left(T^{\mathrm{SMU}}_{\mathrm{Transfer}} - T^{\mathrm{SMU}}_{\mathrm{Nat.\,ref.}}\right) = \quad (1.)$$

$$(78.9\ \mu\mathrm{K}) + (6.7\ \mu\mathrm{K}) + (-\ 81.4\ \mu\mathrm{K}) = 4.2\ \mu\mathrm{K}$$

Uncertainty $u(T^{\mathrm{CMI}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}})$ was calculated as:

$$u^2\left(T^{\mathrm{CMI}}_{\mathrm{Nat.\,ref.}} - T^{\mathrm{SMU}}_{\mathrm{Nat.\,ref.}}\right) = u^2\left(T^{\mathrm{CMI}}_{\mathrm{Nat.\,ref.}} - T^{\mathrm{CMI}}_{\mathrm{Transfer}}\right) + u^2\left(T^{\mathrm{CMI}}_{\mathrm{Transfer}} - T^{\mathrm{SMU}}_{\mathrm{Transfer}}\right) + u^2\left(T^{\mathrm{SMU}}_{\mathrm{Transfer}} - T^{\mathrm{SMU}}_{\mathrm{Nat.\,ref.}}\right) = \quad (2.)$$

$$(60\ \mu\mathrm{K}) + (29\ \mu\mathrm{K}) + (-\ 43\ \mu\mathrm{K}) = 79\ \mu\mathrm{K}$$

The temperature difference $T^{\mathrm{CMI}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{CMI}}_{\mathrm{Transfer}}$. with associated combined standard uncertainty ($k = 1$) is:

$$T^{\mathrm{CMI}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{CMI}}_{\mathrm{Transfer}} = 4.2\ \mu\mathrm{K}$$

$$u\,(T^{\mathrm{CMI}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{CMI}}_{\mathrm{Transfer}}) = 79\ \mu\mathrm{K}$$

5 Linkeage to EUROMET.T-K7

The linkage to EUROMET.T-K7 (T_{ERV}) for the SMU was obtained from the Table XX [1].

$$T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}} - T_{ERV} = \left(T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{PREV}}_{\mathrm{SMU\ Nat.\ ref.}}\right) + \left(T^{\mathrm{PREV}}_{\mathrm{SMU\ Nat.\ ref.}} - T_{ERV}\right) = \quad (3.)$$

$$(6.5\ \mu\mathrm{K}) + (-19\ \mu\mathrm{K}) = -12.5\ \mu\mathrm{K}$$

$$u^2\left(T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}} - T_{ERV}\right) = u^2\left(T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{PREV}}_{\mathrm{SMU\ Nat.\ ref.}}\right) + u^2\left(T^{\mathrm{PREV}}_{\mathrm{SMU\ Nat.\ ref.}} - T_{ERV}\right) = \quad (4.)$$

$$(42\ \mu\mathrm{K})^2 + (59\ \mu\mathrm{K})^2$$

$$u\left(T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}} - T_{ERV}\right) = \quad 72\ \mu\mathrm{K}$$

The linkage to EUROMET.T-K7 (T_{ERV}) for the CMI is provided through comparison of CMI national reference and actual SMU national reference.

$$T^{\mathrm{CMI}}_{\mathrm{Nat.\ ref.}} - T_{ERV} = \left(T^{\mathrm{CMI}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}}\right) + \left(T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}} - T_{ERV}\right) =$$
$$\left[\left(T^{\mathrm{CMI}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{CMI}}_{\mathrm{Transfer}}\right) + \left(T^{\mathrm{CMI}}_{\mathrm{Transfer}} - T^{\mathrm{SMU}}_{\mathrm{Transfer}}\right) + \left(T^{\mathrm{SMU}}_{\mathrm{Transfer}} - T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}}\right)\right] +$$
$$\left[\left(T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{PREV}}_{\mathrm{SMU\ nat.\ ref.}}\right) + \left(T^{\mathrm{PREV}}_{\mathrm{SMU\ nat.\ ref.}} - T_{ERV}\right)\right] = \quad (5.)$$

$$(78.9\ \mu\mathrm{K}) + (6.7\ \mu\mathrm{K}) + (-81.4\ \mu\mathrm{K}) + (6.5\ \mu\mathrm{K}) + (-81.4\ \mu\mathrm{K}) = 8.3\ \mu\mathrm{K}$$

$$u^2\left(T^{\mathrm{CMI}}_{\mathrm{Nat.\ ref.}} - T_{ERV}\right) = u^2\left(T^{\mathrm{CMI}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}}\right) + u^2\left(T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}} - T_{ERV}\right) =$$
$$\left[u^2\left(T^{\mathrm{CMI}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{CMI}}_{\mathrm{Transfer}}\right) + u^2\left(T^{\mathrm{CMI}}_{\mathrm{Transfer}} - T^{\mathrm{SMU}}_{\mathrm{Transfer}}\right) + u^2\left(T^{\mathrm{SMU}}_{\mathrm{Transfer}} - T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}}\right)\right] +$$
$$\left[u^2\left(T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{PREV}}_{\mathrm{SMU\ nat.\ ref.}}\right) + u^2\left(T^{\mathrm{PREV}}_{\mathrm{SMU\ nat.\ ref.}} - T_{ERV}\right)\right] = \quad (6.)$$

$$[(60\ \mu\mathrm{K})^2 + (29\ \mu\mathrm{K})^2 + (43\ \mu\mathrm{K})^2] + [(21\ \mu\mathrm{K})^2 + (59\ \mu\mathrm{K})^2)]$$

$$u\left(T^{\mathrm{CMI}}_{\mathrm{Nat.\ ref.}} - T_{ERV}\right) = \quad 101\ \mu\mathrm{K}$$

Comment: *uncertainty of* $\left(T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}} - T^{\mathrm{PREV}}_{\mathrm{SMU\ Nat.\ ref.}}\right)$ was taken a*s 21 μK, to exclude double counting of uncertainty for* $T^{\mathrm{SMU}}_{\mathrm{Nat.\ ref.}}$ *for the CMI (see Table 4.)*

6 Immersion profiles

CMI were asked to provide an immersion profile of their transfer cell. The result is shown in the following graphs. The slope of the linear fit to the data obtained from CMI transfer cell was stated $-0.64 \cdot 10^{-3}$ K/m.

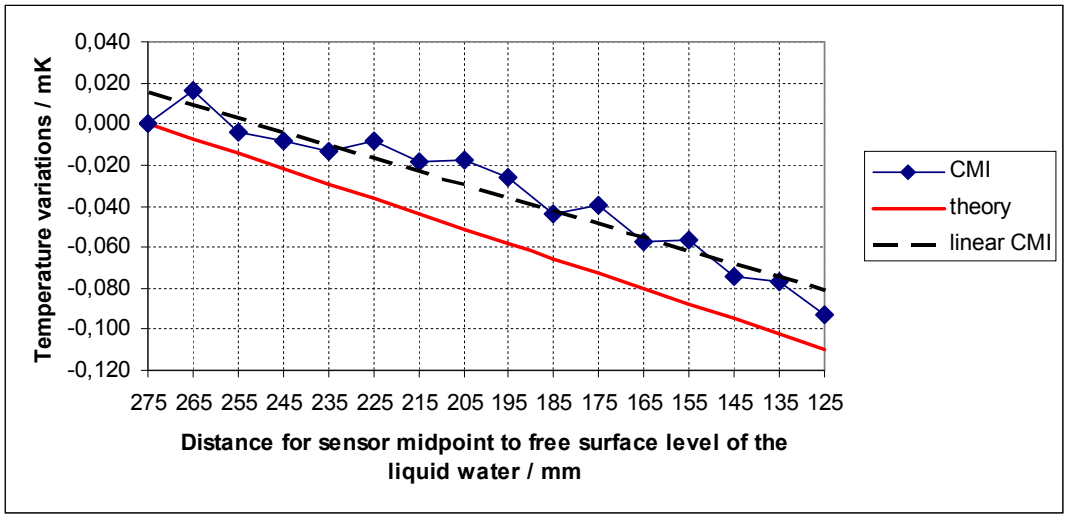

Fig. 2: Immersion profile obtained from CMI transfer cell.

Reference cell	*T* (Reference cell)- *T* (ERV) / µK	*U*(k=2) / µK
SMU Nat.ref.(previous)	-19	118
SMU Nat.ref.(actual)	-12.5	144
CMI Nat.ref.	8.3	202

Table 13: Degrees of equivalence and associated uncertainty (k = 2).

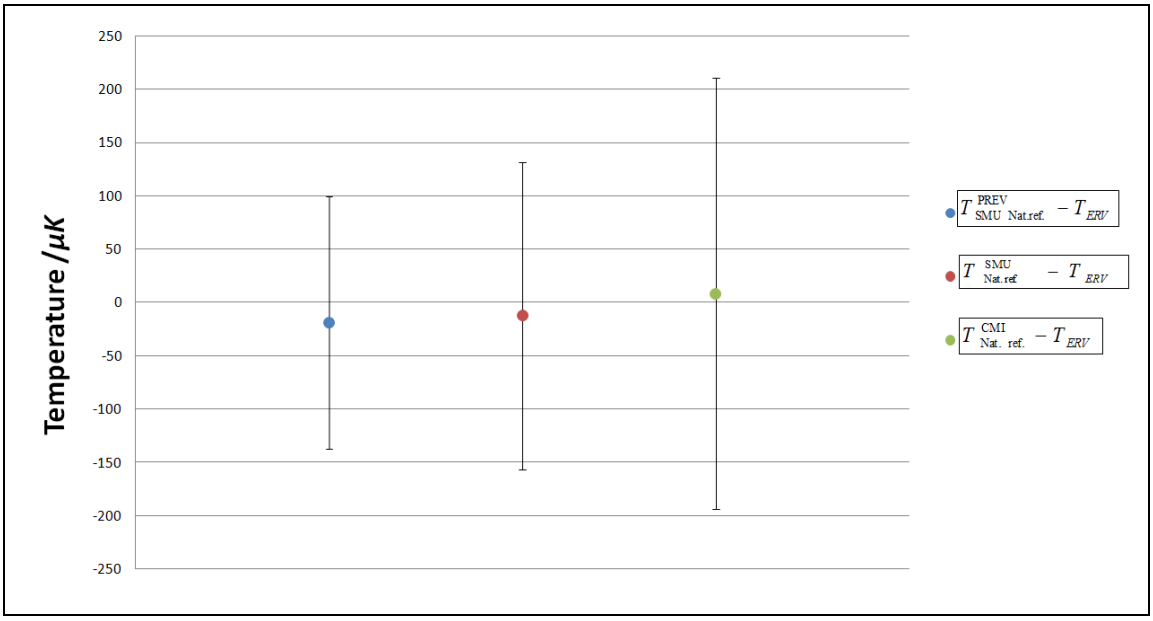

Fig. 3: Temperature differences between the EURAMET.T-K7 , SMU and CMI national cells. Uncertainty bars at k = 2.

Reference

[1] *Report to the CCT on Key Comparison EUROMET.T-K7(EUROMET Project 899), Final Report, November 2008.*