

EURAMET Project n. 1163

**Bilateral comparison of water
triple point cells:
EURAMET.T-K7.2**

Prepared by:

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FINAL REPORT

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1. ORGANIZATION OF THE COMPARISON

1.1 Introduction

The Central Office of Measures, GUM, as the official signatory of the CIPM MRA for Poland, designated the Institutes of Low Temperature and Structure Research, INTiBS, for participation in the CIPM MRA in the low temperature range of the thermometry field. The triple point of water (TPW) temperature is the border between the low temperature range (covered by INTiBS) and the high temperature range (covered by GUM). GUM and INTiBS agreed that GUM provides the services at the TPW related to the use of long-stem SPRTs (LSPRTs) while INTiBS provides the services at the TPW related to the use of capsule-type SPRTs (CSPRTs). Only GUM took part in EURAMET.T-K7 comparison of water triple point cells. As consequence INTiBS requested a bilateral comparison with VSL (pilot of EURAMET.T-K7) to:

- demonstrate its technical competence in calibrating CSPRTs at the TPW
- support CMCs for CSPRTs at the TPW and publish them in the KCDB.

Following the nomenclature of BIPM, this bilateral comparison was named EURAMET.T-K7.2.

1.2 Objectives of the comparison

The specific objectives of this comparison were:

- 1) A direct comparison of TPW cells to quantify the differences between the TPW temperatures realized by the cells
- 2) A comparison of the national realizations of the TPW temperature.

1.3 Participants

The participant laboratories with corresponding contact details are listed in the Table 1:

Acronym	Institute	Country	Contact person and details
VSL	VSL, Dutch Metrology Institute	The Netherlands	Andrea Peruzzi aperuzzi@vsl.nl +31 15 269 1519
INTiBS	Institute of Low Temperature and Structure Research	Poland	Anna Szmyrka-Grzebyk A.Szmyrka@int.pan.wroc.pl +48 71 3954 264

Table 1: Participants and contact details

1.4 Comparison method

The pilot laboratory for this comparison was VSL. The measurement pattern was the following:

- **Part 1:** INTiBS selected its transfer cell and compared it to its national reference (either single cell or group of cells) by means of CSPRTs
- **Part 2:** INTiBS transported its transfer cell to VSL. VSL compared INTiBS transfer cell to VSL transfer cell and to VSL national reference by means of LSPRTs
- **Part 3:** INTiBS transported back its transfer cell and compared it again to its national reference.

1.5 Transfer cells, national reference cells and national reference at VSL

At VSL the national reference for the TPW temperature is defined by the average of 12 well-characterized TPW cells (see Table 2). These are the same 12 cells that served as VSL national reference in EURAMET.T-K7. In the second column of Table 2, the temperature difference between each cell of the national reference and cell VSL98T094, which is the VSL transfer cell in CCT-K7 and EURAMET.T-K7, is given (no isotopic correction applied). In 2006, after the clarification of the definition of the Kelvin and extensive internal investigation at VSL of the isotopic composition of the reference cells, VSL shifted its national reference by 73 μK up with respect to the VSL national reference in CCT-K7. In the third column, the difference between the VSL transfer cell in both CCT-K7 and EURAMET.T-K7 and the new VSL national reference (from 2006 on) is given. In the last column, the difference between each cell of the VSL national reference and the new VSL national reference is given. For this comparison cell VSL06T003 and VSL06T004 were used (see the two rows in bold characters in Table 2).

Cell identification	$(T_i - T_{\text{VSL98T094}})/\mu\text{K}$	$(T_{\text{VSL98T094}} - T_{\text{Nat. Ref}})/\mu\text{K}$	$(T_i - T_{\text{Nat. Ref}})/\mu\text{K}$
VSL03T026	-27	-14	-41
VSL03T028	-20	-14	-34
VSL03T029	-30	-14	-44
VSL03T030	-26	-14	-40
VSL03T032	-33	-14	-47
VSL03T039	-61	-14	-75
VSL06T001	-19	-14	-33
VSL06T002	-13	-14	-27
VSL06T003	+1	-14	-13
VSL06T004	-4	-14	-18
VSL06T006	-14	-14	-28
VSL06T007	-9	-14	-23

Table 2: Batch of 12 cells defining the VSL national reference. The second column shows the temperature differences between each cell and the VSL transfer cell used in CCT-K7 and EURAMET.T-K7. The last column shows the temperature difference between each cell and the new VSL national reference (from 2006 on).

1.6 Transfer cells, national reference cells and national reference at INTiBS

At INTiBS the national reference is defined by a single cell: ISOTECH type B11-50-270, serial n. 901 of known isotopic composition. The transfer cell selected by INTiBS was ISOTECH type B11-50-270, serial n. 899.

Table 3 contains an overview of the information available on the transfer and reference cells used in this comparison.

Lab.	Transfer cell			National reference cell(s)				
	Serial n.	Manufacturer /model	Year	Definition	Serial n.	Manufacturer /model	Year	Isotopic composition
VSL	VSL06T003 VSL06T004	VSL	2006	Group of 12 cells	VSL06T003 VSL06T004	VSL		
INTiBS	899	ISOTECH B11-50-270	2010	Single cell	901	ISOTECH B11-50-270		$\delta^{18}\text{O} = 1.011 \text{ ‰}$ $\delta^2\text{H} = 12.7 \text{ ‰}$

Table 3: Overview information available information on transfer and reference cells.

2. EQUIPMENT, TECHNIQUES AND UNCERTAINTIES

2.1 Laboratory equipment and techniques

The equipment, the techniques and the measuring conditions at the two participating laboratories are summarized in Table 4.

In the measurements performed at INTiBS, the capsule SPRT was accommodated in a copper bushing inside the thermometer well. The liquid used in the thermometer well was Kryo-85 oil. The electrical leads to the capsule SPRT were lacquer and silk-insulated tin-copper wires. Above the oil surface, the wires were inserted in a Teflon tube. The resistance of the CSPRT was measured at three different currents: 1 mA, 1.42 mA and 2 mA. The zero current resistance was then obtained by extrapolation. A hydrostatic correction was also applied.

	INTiBS	VSL
Resistance ratio bridge	MI 6015T (DC)	MI 6015T (DC)
Measurement current(frequency)	1 mA, $\sqrt{2}$ mA and 2 mA(DC)	2 and $2\sqrt{2}$ mA (DC)
Number of repeated measurements	40	25
Frequency of repeated measurements	0.1 Hz	0.1 Hz
Reference resistor	25 Ω , Tinsley, type 5685A	25 Ω , Tinsley, type 5684S
Temperature control of reference resistor	± 30 mK	± 1 mK
SPRT and sensor length	CSPRT Rosemount 25 Ω	LSPRT L&N, Cat. 8167-25 (33 mm)
Storage container for TPW cells	Isotech water triple point bath	Isotech 18233
Technique ice mantle preparation	LN cooler bar	LN flow-through cooling

Table 4: Overview equipment, techniques and measuring conditions at the participating laboratories.

2.2 Uncertainties

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

Origin	INTiBS	VSL
Comparison of transfer cell and national reference (components related only to the comparison of the two cells)		
Repeatability for a single ice mantle (including bridge noise)	7	11
Reproducibility for different ice mantles	20	8
Reproducibility for different types of SPRTs	-	
Hydrostatic head of transfer cell	2	5
Hydrostatic head of reference cell	2	
SPRT self-heating in the transfer cell and reference cell	5	7
Perturbing heat exchanges	4	5
Differential bridge non-linearities	0.3	6
National reference (components related only to properties of the reference cell(s))		
Chemical impurities	20	20
Isotopic variation	3	2
Residual gas pressure in the cell	5	3
Reproducibility	20	5
Total uncertainty (k=1)	37	28

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

3. MEASUREMENTS AT INTiBS

The measurements at INTiBS were performed from 20/09/2010 to 22/10/2010. The results of the measurements are summarized in Table 6.

Measurement n.	First ice mantle	Second ice mantle
	$(T_{transfer}^{INTiBS} - T_{Nat. Ref.}^{INTiBS}) / \mu K$	$(T_{transfer}^{INTiBS} - T_{Nat. Ref.}^{INTiBS}) / \mu K$
1	4	-23
2	46	9
3	15	-35
4	7	20
5	15	-15
6	14	8
7	-6	1
8	-10	-3
9	-43	-29
10	-16	6
11	1	-
Mean	2.5	-6.1
Standard deviation of the mean	6.7	5.8

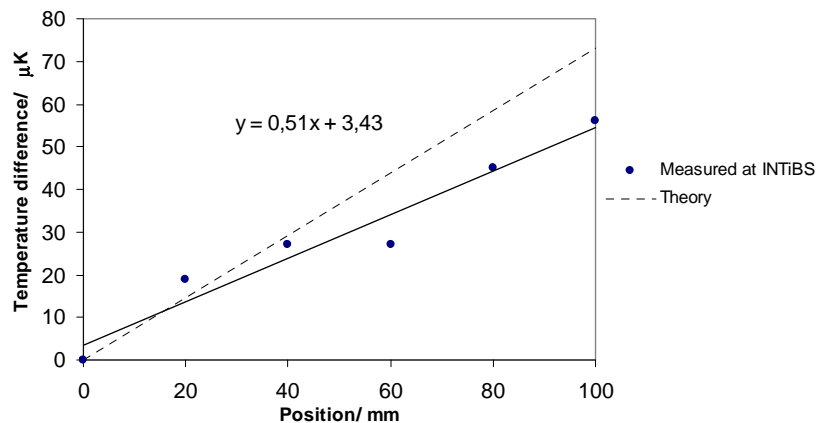
Table 6: Overview results at INTiBS.

$$T_{transfer}^{INTiBS} - T_{Nat. Ref.}^{INTiBS} = -1.8 \mu K$$

$$u(T_{transfer}^{INTiBS} - T_{Nat. Ref.}^{INTiBS}) = 37 \mu K (k = 1)$$

Immersion profile of transfer cell (measured by INTiBS)

Distance from sensor midpoint to free surface level of the liquid water, mm	Temperature variation, μK
0	0
20	19
40	27
60	27
80	45
100	56



4. MEASUREMENTS AT VSL

At VSL the transfer cell of INTiBS (cell n. 899) was compared to VSL cells VSL06T003 and VSL06T004. From comprehensive internal comparisons at VSL, the triple point temperatures realized by the cells VSL06T003 and VSL06T004 relative to the VSL national reference for the triple point of water temperature are known to be (see Table 2):

$$T_{VSL06T003} - T_{Nat. Ref.}^{VSL} = -12.7 \mu\text{K}$$

$$u(T_{VSL06T003} - T_{Nat. Ref.}^{VSL}) = 28 \mu\text{K} (k = 1)$$

$$T_{VSL06T004} - T_{Nat. Ref.}^{VSL} = -17.7 \mu\text{K}$$

$$u(T_{VSL06T004} - T_{Nat. Ref.}^{VSL}) = 28 \mu\text{K} (k = 1)$$

Accordingly, the VSL national reference in this comparison was built up directly from the temperatures realized by VSL cells VSL06T003 and VSL06T004 as:

$$T_{Nat. Ref.}^{VSL} = [(T_{VSL06T003} + 12.7 \mu\text{K}) + (T_{VSL06T004} + 17.7 \mu\text{K})]/2$$

The measurements at VSL were performed from 29/11/2010 to 31/12/2010. The results are summarized in Table 7.

Measurement n.	First ice mantle	Second ice mantle
	$(T_{transfer}^{INTiBS} - T_{Nat. Ref.}^{VSL})/ \mu\text{K}$	$(T_{transfer}^{INTiBS} - T_{Nat. Ref.}^{VSL})/ \mu\text{K}$
1	-12.3	4.4
2	24.1	10.8
3	-6.9	8.4
4	-2.4	17.7
5	-15.7	12.8
6	-8.3	6.4
7	-7.3	17.7
8	-7.8	-1.0
9	-17.7	3.0
10	-	0.5
Mean	-6.0	8.1
Standard deviation of the mean	4.1	2.1

Table 7: Overview results at VSL.

$$T_{transfer}^{INTiBS} - T_{Nat. Ref.}^{VSL} = 1.1 \mu\text{K}$$

$$u(T_{transfer}^{INTiBS} - T_{Nat. Ref.}^{VSL}) = 28 \mu\text{K} (k = 1)$$

5. PAIR EQUIVALENCE BETWEEN INTiBS AND VSL

The bilateral equivalence between INTiBS and VSL was found as:

$$T_{Nat. Ref.}^{INTiBS} - T_{Nat. Ref.}^{VSL} = (T_{Nat. Ref.}^{INTiBS} - T_{Transfer Cell}^{INTiBS}) + (T_{Transfer Cell}^{INTiBS} - T_{Nat. Ref.}^{VSL}) = 2.9 \mu\text{K}$$

$$u^2(T_{Nat. Ref.}^{INTiBS} - T_{Nat. Ref.}^{VSL}) = u^2(T_{Nat. Ref.}^{INTiBS} - T_{Transfer Cell}^{INTiBS}) + u^2(T_{Transfer Cell}^{INTiBS} - T_{Nat. Ref.}^{VSL}) = (37^2 + 28^2) (\mu\text{K})^2 = 46 \mu\text{K} (k = 1)$$

6. LINKAGE TO EURAMET.T-K7

The linkage to EURAMET.T-K7 is provided directly by the pair equivalence between INTiBS and VSL found in the previous section.

Remembering that:

- the DoE of VSL in EURAMET.T-K7 was:
 $D_{VSL} = T_{VSL} - T_{ERV} = -1.7 \mu\text{K}$
 $U_{VSL} = 60 \mu\text{K}$
- the pair equivalence between INTiBS and VSL established in this comparison is (see section 5):
 $D_{INTiBS, VSL} = T_{INTiBS} - T_{VSL} = +2.9 \mu\text{K}$
 $U_{INTiBS, VSL} = 93 \mu\text{K}$

The DoE of INTiBS in EURAMET.T-K7 is found as:

$$D_{INTiBS} = (T_{INTiBS} - T_{VSL}) + (T_{VSL} - T_{ERV}) = +2.9 \mu\text{K} + (-1.7 \mu\text{K}) = 1.2 \mu\text{K}$$

$$U_{INTiBS} = [(U_{INTiBS, VSL})^2 + (U_{VSL})^2]^{1/2} = [(93 \mu\text{K})^2 + (60 \mu\text{K})^2]^{1/2} = 111 \mu\text{K}$$

The pair equivalence between INTiBS (LAB_i) and any other EURAMET.T-K7 participant LAB_j is calculated as (see Table 8):

$$D_{INTiBS, LAB_j} = D_{INTiBS} - D_{LAB_j}$$

$$U_{INTiBS, LAB_j} = [(U_{INTiBS, VSL})^2 + (U_{VSL, LAB_j})^2]^{1/2}$$

$LAB_j (LAB_i = INTiBS)$	$D_{ij} / \mu\text{K}$	$U_{ij} / \mu\text{K}$
CEM	-34.4	143
INM	+19.7	171
MKEH	+94.2	178
INRiM	+15.0	127
EIM	-28.6	253
MIKES	+33.4	172
PTB	-22.5	135
LNE-INM/CNAM	+0.9	159
DTI	+68.0	178
JV	+162.9	404
VNIIM	-46.3	153
MIRS/FE-LMK	-16.2	124
DZM-LPM	+7.2	151
UME	+102.6	191
ZMDM	+167.5	264
SMD	-27.6	132
BEV	+37.2	366
IPQ	-12.8	196
NML	-23.1	241
SMU	+20.7	162
CMI	+327.0	166
GUM	+325.7	188
VMT/PFI	+79.2	264
VSL	+2.9	93

Protocol of Measurements
EURAMET.T-K7.2
Comparison of water triple point cells

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Draft B (16/09/10)

1. Introduction

GUM, as the official signatory of the CIPM MRA for Poland, designated INTiBS for participation in the CIPM MRA in the low temperature range of the thermometry field. The water triple point temperature is the border between the low temperature range (covered by INTiBS) and the high temperature range (covered by GUM). GUM and INTiBS agreed that GUM provides the services at the water triple point related to the use of long-stem SPRTs (LSPRTs), while INTiBS provides the services at the water triple point related to the use of capsule-type SPRTs (CSPRTs). Only GUM took part in EURAMET.T-K7 of water triple point cells. As consequence INTiBS required a comparison with VSL (pilot of EURAMET.T-K7) to demonstrate its technical competence in calibrating CSPRTs at the triple point of water. Following the nomenclature of BIPM comparison, this comparison was named EURAMET.T-K7.2.

This technical protocol describes the objectives of the EURAMET.T-K7.2 comparison, its organization and procedures to be followed by the participants. It has been drawn up according to the following documents:

- The technical supplement to the CIPM document “*Mutual Recognition Arrangement of national measurement standards and of calibration and measurement certificates issued by national metrology institutes (MRA)*” [1].
- The BIPM document “*Guidelines for CIPM key comparison*” [2].
- The EUROMET guide n. 3 “*Euromet Guidelines on Conducting Comparisons*” [3].

To ensure proper link to its corresponding CIPM key comparison, this technical protocol is in most of its parts very similar to that already used for CCT-K7 and EURAMET.T-K7.

All participants to this comparison accept the general instructions and commit themselves to follow the procedures described in this technical report. Once the protocol has been agreed, no change to the protocol or to the list of participants may be accepted without prior agreement of all the participants.

2. Objectives of the comparison

The specific objectives of this comparison are:

- 1) A direct comparison of water triple point cells to quantify the differences between the cells.
- 2) A comparison of the national realizations of the WTP temperature.

3. Organization of the comparison

The participants with corresponding contact person are listed in the following table:

Acronym	Contact person	Country
INTiBS	Anna Szmyrka-Grzebyk	Poland
VSL	Andrea Peruzzi	The Netherlands

Table 1: List of participants with corresponding contact persons.

The pilot laboratory for this comparison is VSL.

The measurement pattern can be essentially divided into three parts:

- **Part 1:** INTiBS selects its *transfer cell* and compares it to its *national reference* (either single cell or group of cells) **by means of capsule SPRTs (CSPRTs)**
- **Part 2:** INTiBS transports its *transfer cell* to VSL. VSL compares INTiBS *transfer cell* to VSL *transfer cell* and VSL *national reference* by means of long-stem SPRTs (LSPRTs).
- **Part 3:** INTiBS transports back its *transfer cell* and compares it again to its *national reference*.

The transport of the transfer cell from INTiBS to VSL and back from VSL to INTiBS is within the responsibility of INTiBS.

The cells should be hand-carried. The cells must be accompanied by an ATA carnet or a temporary export document (where appropriate). Also an eventual insurance of the cells for the transport is within the responsibility of INTiBS. Before dispatching the cell, INTiBS shall inform VSL. After arrival of the cell, VSL shall inform INTiBS and, immediately after receipt, VSL shall check for any damage of the cell and report it to the pilot institute. If INTiBS uses special parts with its cell, like a bushing or a foam pad, these should also be sent to VSL, together with the description of its use (if necessary).

The timetable of the comparison is the following:

- 21/09/10: Starting date
- 21/09/10 to 30/10/10: INTiBS compares its *transfer cell* to its *national reference* (either single cell or group of cells) and transports its *transfer cell* to VSL.
- 30/10/10: Deadline transport of INTiBS *transfer cell* to VSL and delivery of the results of INTiBS measurements.
- 30/10/10 to 10/12/10: VSL compares INTiBS *transfer cell* to VSL *transfer cell* and VSL *national reference*.
- 10/12/10: Deadline measurements at VSL
- 10/12/10 to 31/01/11: INTiBS transports back its *transfer cell* and compares it again to its *national reference*.
- 31/01/11: Deadline delivery INTiBS results of return measurements.

5. Selection of the transfer cells

VSL will use as transfer cell VSL094, the same used in both CCT-K7 and EURAMET.T-K7.

The cell chosen by INTiBS as its transfer cells for this comparison should be carefully selected. The quality of the transfer cell should not significantly differ from the reference cell or cells used at INTiBS. No cell must be used whose quality is suspect on simple inspection procedures or which is known for any kind of abnormal behavior.

The following tests should be made on the INTiBS transfer cell and will be repeated at reception of the cell at VSL:

- No floating material should be visible in the water.
- There should be a sharp “click” audible if the cell is gently inverted, indicating very low amount of residual air (“water hammer test”).
- For the cells where it is possible, a McLeod type test should be made by inverting the cell and entrapping air in the side arm or in the filling extension. The allowable bubble size for an acceptable cell depends on the cell type. For example, for a Jarrett Type A cell, the bubble diameter should not be larger than about 5 mm, corresponding to a temperature depression of 5 μ K. Prior to testing for air, the cells should be held vertically at room temperature overnight.

VSL reserves the right to reject transfer cells that do not meet the minimum selection criteria when tested on receipt. If INTiBS is normally using other tests, INTiBS is invited to apply them in addition and to describe them.

INTiBS is asked to provide as soon as possible information about the dimensions (in cm) of the selected transfer cell. This particularly applies to cells with unusual dimensions (for examples, very large or very small cells).

6.1. Measurements instructions and reporting

Each laboratory must carefully select its transfer cell according to the criteria given in paragraph 5 and compare it against its national reference (single cell or set of cells). The measurements should be performed on two separately prepared ice mantles of the transfer cell and, for each prepared ice mantle, the direct comparison to the national reference should be finalized within two weeks with typically one measurement per day. The measurements should not start earlier than 7 days after the preparation of the ice mantle. Depending on the local preparation technique, the minimum waiting time required might be longer than 7 days. A minimum of 10 measurements per mantle should be reported in the appropriate Measurement Report Form A (see appendix 1). Before each measurement an inner melt shall be induced. The recommended method for inducing the inner melt is the insertion of a room temperature metal or glass rod in the thermometer well for a few seconds. The ice mantle should then be freely rotating around the well when a small rotational impulse is given to it. The well should be filled with pre-cooled pure water up to the level of the water in the cell, when the thermometer is in place. To reduce the transfer uncertainty, the participants might consider preparing the ice mantle of the transfer cell by using the same technique adopted by the assigned co-pilot. In this case, the two parts will have to arrange themselves the needed exchange of information. Apart from this, the measurement procedure should be the one normally applied by the laboratory.

For each transfer cell, an immersion profile should be provided, to ensure that the measurement really senses the temperature of the ice/water interface. For each

position, the self-heating correction should be determined and applied. The step width should be 1 or 2 cm, and the measurements should be taken up to about 10 cm below the water surface. The position of the sensor at which the comparison with the reference cell(s) was made should be indicated.

After its return from VSL laboratory, the stability of the INTiBS transfer cell must be checked with an additional comparison against the INTiBS national reference. The full set of measurements described above should be repeated.

If the cell is found to be stable, this information should be given to the coordinator; in this case only the measurements made before sending the cell to VSL will be used. If a small, but significant drift is discovered, the laboratory should send the new results (within 3 months after receiving back the transfer cell) to the coordinator, in the same form as before and a new final value for the temperature of the cell can be determined, based on all measurements. If a cell is found unstable ($\Delta T > 100 \mu\text{K}$, or criteria identified by the participant before the comparison begins), the laboratory should inform VSL as early as possible, and within 6 months. In case the feedback measurement is not provided within the time foreseen, only the first result will be used for the data analysis.

6.2. Reporting the measurement results

Each laboratory must report the performed measurements by filling the appropriate Measurement Report Forms. The Measurement Report Forms shown in the Appendixes will be made available in the form of Excel sheets to be filled by the participants. The reporting of the measurement results will include at least the following:

- The daily results obtained during the two measurement phases on the two separately prepared mantles. The self-heating (0 mA) and hydrostatic head correction (immersion depth) should be applied to the results, and the corrections for the transfer cell also communicated separately. Based on these data sets a resulting temperature difference of the transfer cell from the national reference has to be stated.

- The immersion profile of the transfer cell, indicating the position of the sensor at which the calibration was made.

- A detailed budget for the uncertainty of the temperature realized by the transfer cell has to be provided, which follows the general guidance of the 'GUM' [7]. This budget shall include the uncertainty of the national standard (realization uncertainty) and of the direct comparison of the transfer cell to the standard. A model uncertainty budget is given in the Appendix 1.

- The equipment used for the calibration: description of the national reference, technique to prepare the ice mantel, type of storage container, type of thermometer, type of resistance bridge (AC or DC), type of reference resistor and whether or not it is temperature controlled, purchase or manufacturing date of reference cell(s) and transfer cell, measurement currents, and age of mantles of the reference cell(s). If available, the results of an isotope and/or impurity analysis.

7. General rules of the comparison

To resolve problems concerning eventual incomplete or anomalous data, the general rules of the guidelines for CIPM key comparison [2] will be applied. The full text can be found on the BIPM web page (www.bipm.fr/pdf/guidelines.pdf), and in the following we give an extract of some rules that are the most important according to our experience:

- During the comparison, as the results are received by the pilot institute, they are kept confidential by the pilot institute until all the participants have completed their measurements and all the results have been received, or until the date limit for receipt of results has passed.
- A result from a participant is not considered complete without an associated uncertainty, and is not included in the draft report unless it is accompanied by an uncertainty supported by a complete uncertainty budget. Uncertainties are drawn up following the guidance given in the technical protocol.
- If, on examination of the complete set of results, the pilot institute finds results that appear to be anomalous, the corresponding institutes are invited to check their results for numerical errors but without being informed as to the magnitude or sign of the apparent anomaly. If no numerical error is found the result stands and the complete set of results is sent to all participants. Note that once all participants have been informed of the results, individual values and uncertainties may be changed or removed, or the complete comparison abandoned, only with the agreement of all participants and on the basis of a clear failure of the travelling standard or some other phenomenon that renders the comparison or part of it invalid.
- An institute that considers its result unrepresentative of its standards may request a subsequent separate bilateral comparison with the pilot institute or one of the participants. This should take place as soon as possible after the completion of the comparison in progress. The subsequent bilateral comparison is considered as a new and distinct comparison (see paragraph 10).

It is difficult to give in advance an unambiguous criterion for what constitutes anomalous data. The pilot will consider this depending on the real data. Data, which according to common sense would be called an outlier, will be considered as anomalous and the corresponding laboratory will be asked to verify its calculation. In case of any doubt we will contact the corresponding laboratory.

8. References

- [1] “*Mutual Recognition Arrangement of national measurement standards and of calibration and measurement certificates issued by national metrology institutes (MRA)*”, BIPM, 1999.
- [2] T. J. Quinn, “*Guidelines for CIPM key comparison*”, 1 March 1999, BIPM, Paris.
- [3] EUROMET Guide n. 3, “*Euromet Guidelines on Conducting Comparisons*”, version 02.7.
- [4] “*Summary of Facts Relating to Isotopic Effects and the Triple Point of Water: report of the ad hoc Task Group on the triple point of water*”, CCT document CCT/05-07.
- [5] “*Methodologies for the estimation of the uncertainties and the correction of fixed point temperatures attributable to the influence of chemical impurities*”, the CCT document CCT/05-08.
- [6] CCT WG8 report to the 23rd CCT Meeting (2005).
- [7] “*Guide to the expression of uncertainty in measurement*”, ISO, 1993, ISBN 92-67-10188-9.

Appendix 1: Measurement Report Form A (for INTiBS, to be used in both Part 1 and Part 3)

Laboratory:

Contact person:

Contact address and e-mail:

Transfer cell (n° and type):

Purchase or manufacture date:

Measurement results on first ice mantle

Date of preparation of first ice mantle of transfer cell:

Technique for preparation:

Date of preparation of the mantle of the reference cell(s):

Date of measurement	Temperature difference from national reference	Experimental standard deviation of temperature difference from national reference	Distance from sensor midpoint to surface level of water in transfer cell	Self-heating correction for transfer cell
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				

The temperature differences should already be corrected for hydrostatic-head and self-heating effects for both the national reference and the transfer cell. If sufficient information is available on the cell (group of cells) defining the national reference, an isotopic correction can be applied to the national reference. **No isotopic correction must be applied to the transfer cell.**

Measurement results on second ice mantle

Date of preparation of second ice mantle:

Technique for preparation:

Date of preparation of the mantle of the reference cell(s):

Date of measurement	Temperature difference from national reference	Experimental standard deviation of temperature difference from national reference	Distance from sensor midpoint to surface level of water in transfer cell	Self-heating correction for transfer cell
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				

The temperature differences should already be corrected for hydrostatic-head and self-heating effects for both the national reference and the transfer cell. If sufficient information is available on the cell (group of cells) defining the national reference, an isotopic correction can be applied to the national reference. **No isotopic correction must be applied to the transfer cell.**

Resulting temperature difference between transfer cell and national reference:

Equipment used for the measurements

Description of national reference (1 or several cells, purchase or manufacture date):

Type of resistance bridge, AC or DC:

Measurement current:

Number and sampling frequency of repeated measurements:

Type of reference resistor:

Is reference resistor temperature-controlled? (If yes, state stability):

Type of thermometer, length of sensor:

Storage container for WTP cells:

Immersion profile (only for transfer cell)

Distance from sensor midpoint to free surface level of the liquid water	Temperature variation

The above table is for reporting measurement of the hydrostatic head effect. Measurements should be taken at a step width of 1 to 2 cm. Thermometer readings should be corrected for self-heating, measured at each position.

Uncertainty Budget

The uncertainty budget should include the components listed on the table, to which others can be added if necessary. The uncertainty budget shown here can only be considered as a model. Additional uncertainty components can be added reflecting the peculiarities of each participant laboratory. Please explain, how the contributions of chemical impurities and isotope variation were evaluated.

The repeatability for a single ice mantle is understood as the experimental standard deviation of the daily obtained temperature differences between the transfer cell and the national reference, divided by the square root of the number of daily results (here typically 10). The reproducibility for different ice mantles represents the additionally variability introduced by measuring on several different ice mantles.

All contributions should be stated at the level of one standard uncertainty.

Origin	Contribution (k=1)
National reference (Uncertainties related only to properties of the reference cell)	
Chemical impurities (please explain how estimated)	
Isotopic variation (please explain how estimated)	
Residual gas pressure in cell	
Reproducibility [1]	
Comparison of transfer cell to national reference (Uncertainties related to the comparison of the two cells)	
Repeatability for a single ice mantel (incl. bridge noise) [2]	
Reproducibility for different ice mantles [3]	
Reproducibility for different types of SPRTs [4]	
Hydrostatic head of transfer cell	
Hydrostatic head of reference cell	
SPRT self-heating in the transfer cell and reference cell [5]	
Perturbing heat exchanges [6]	
others	
Total uncertainty	

[1] Estimate of the reproducibility of the temperature reference due to changes in the following quantities: crystal size, the age of the mantles, different mantles, the handling of the cells before preparation of the mantle.

[2] The repeatability for a single ice mantle is understood as the experimental standard deviation of the daily obtained temperature differences between the transfer cell and the national reference, divided by the square root of the number of daily results (here typically 10). This component takes also in account the stability of reference resistor (temperature effect).

[3] The reproducibility for different ice mantles represents the additional variability introduced by measuring on several different ice mantles on transfer cell (probably the laboratory uses the same ice mantle of the reference cell during the time of measurements).

[4] The observed temperature differences between the transfer and the reference cells could depend on the type of SPRT's. This component takes into account possible SPRT internal insulation leakage.

[5] These uncertainties could be strongly positively correlated. All the measurements are corrected for self-heating effect. If the thermal resistances have approximately the same magnitude in transfer and reference cells the difference between the self-heating corrections is very small. In addition the uncertainties on self-heating corrections in transfer and reference cells are strongly correlated. In this case the uncertainty in self-heating corrections only contributes to the Type A uncertainty of the comparison of the cells.

[6] This component could be estimated:

- by comparing the deviations from expected hydrostatic pressure correction obtained in transfer and reference cells (by changing immersion depth over the length of the sensor ≈ 5 cm).
- by modifying the thermal exchange between thermometer and its environment during the measurements on transfer and reference cells.

Appendix 2: Measurement Report Form B (for VSL, to be used in Part 2)

Laboratory:

Contact person:

Contact address and e-mail:

CCT-K7/EURAMET.T-K7 VSL transfer cell (property, identification number, type, purchase or manufacture date):

INTiBS transfer cell (identification number):

Cell (or group of cells) defining the VSL national reference cells (identification number):

Measurement results on first ice mantle

Date of preparation of first ice mantle of CCT-K7 VSL cell, INTiBS transfer cell and cell (or group of cells) defining the VSL national reference:

Technique for preparation of the ice mantle:

Date of measurement	Temperature difference of INTiBS transfer cell from CCT-K7 VSL cell (and corresponding experimental standard deviation)	Temperature difference of VSL national reference from CCT-K7 VSL cell and corresponding experimental standard deviation)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		

The temperature differences should already be corrected for hydrostatic-head and self-heating effects for both the national reference and the transfer cells. If sufficient information is available on the cell (group of cells) defining the national reference, an isotopic correction can be applied to the national reference. **No isotopic correction must be applied to the transfer cells.**

Measurement results on second ice mantle

Date of preparation of second ice mantle of transfer cell, CCT-K7 VSL cell, INTiBS transfer cell and cell (or group of cells) defining the VSL national reference:

Technique for preparation of the ice mantle:

Date of measurement	Temperature difference of INTiBS transfer cell from CCT-K7 VSL cell (and corresponding experimental standard deviation)	Temperature difference of VSL national reference from CCT-K7 VSL cell and corresponding experimental standard deviation)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		

The temperature differences should already be corrected for hydrostatic-head and self-heating effects for both the national reference and the transfer cells. If sufficient information is available on the cell (group of cells) defining the national reference, an isotopic correction can be applied to the national reference. **No isotopic correction must be applied to the transfer cells.**

Equipment used for the measurements

Description of national reference (1 or several cells, purchase or manufacture date):

Type of resistance bridge, AC or DC:

Measurement current:

Number and sampling frequency of repeated measurements:

Type of reference resistor:

Is reference resistor temperature-controlled? (If yes, state stability):

Type of thermometer, length of sensor:

Storage container for WTP cells:

Immersion profile (one for each measured cell)

Distance from sensor midpoint to free surface level of the liquid water	Temperature variation

The above table is for reporting measurement of the hydrostatic head effect. Measurements should be taken at a step width of 1 to 2 cm. Thermometer readings should be corrected for self-heating, measured at each position.

Uncertainty Budget

The uncertainty budget should include the components listed on the table, to which others can be added if necessary. Since the “CCT guidance document on the uncertainties of SPRT calibrations” of WG 3 does not yet exist and the pilot cannot replace the working group, the budget shown here can only be a model. Some additional guidance can however be obtained from the draft documents [6]. Please explain, how the contributions of chemical impurities and isotope variation were evaluated.

The repeatability for a single ice mantle is understood as the experimental standard deviation of the daily obtained temperature differences between the transfer cell and the national reference, divided by the square root of the number of daily results (here typically 10). The reproducibility for different ice mantles represents the additionally variability introduced by measuring on several different ice mantles.

All contributions should be stated at the level of one standard uncertainty.

Origin	Contribution (k=1)
National reference (Uncertainties related only to properties of the reference cell)	
Chemical impurities (please explain how estimated)	
Isotopic variation (please explain how estimated)	
Residual gas pressure in cell	
Reproducibility [1]	
Comparison of transfer cell to national reference (Uncertainties related to the comparison of the two cells)	
Repeatability for a single ice mantel (incl. bridge noise) [2]	
Reproducibility for different ice mantles [3]	
Reproducibility for different types of SPRTs [4]	
Hydrostatic head of transfer cell	
Hydrostatic head of reference cell	
SPRT self-heating in the transfer cell and reference cell [5]	
Perturbing heat exchanges [6]	
others	
Total uncertainty	

[1] Estimate of the reproducibility of the temperature reference due to changes in the following quantities: crystal size, the age of the mantles, different mantles, the handling of the cells before preparation of the mantle.

[2] The repeatability for a single ice mantle is understood as the experimental standard deviation of the daily obtained temperature differences between the transfer cell and the national reference, divided by

the square root of the number of daily results (here typically 10). This component takes also in account the stability of reference resistor (temperature effect).

[3] The reproducibility for different ice mantles represents the additional variability introduced by measuring on several different ice mantles on transfer cell (probably the laboratory uses the same ice mantle of the reference cell during the time of measurements).

[4] The observed temperature differences between the transfer and the reference cells could depend on the type of SPRT's. This component takes into account possible SPRT internal insulation leakage.

[5] These uncertainties could be strongly positively correlated. All the measurements are corrected for self-heating effect. If the thermal resistances have approximately the same magnitude in transfer and reference cells the difference between the self-heating corrections is very small. In addition the uncertainties on self-heating corrections in transfer and reference cells are strongly correlated. In this case the uncertainty in self-heating corrections only contributes to the Type A uncertainty of the comparison of the cells.

[6] This component could be estimated:

- by comparing the deviations from expected hydrostatic pressure correction obtained in transfer and reference cells (by changing immersion depth over the length of the sensor ≈ 5 cm).
- by modifying the thermal exchange between thermometer and its environment during the measurements on transfer and reference cells.

Appendix 3: Information about the definition of national reference and the link to CCT K7

4.1 For both VSL and INTiBS

Description of national reference (Is the national reference defined by a single cell or a group of cells):

Available information about the isotopic composition of the national reference cell(s):

Available information about the impurity content of the national reference cell(s):

4.2 Only for VSL

Describe in details how the national reference was defined for CCT-K7:

Describe in details how the national reference is defined for this comparison:

Provide details about the magnitude and the technical basis for the eventual change in the definition of the national reference for this comparison with respect to its definition in CCT-K7:

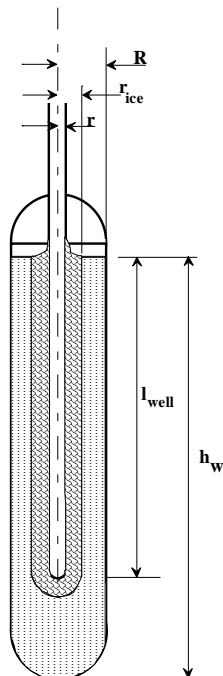


Figure 1: Water triple point cell with ice mantle. R is the radius of the cell, r is the radius of the thermometer well, h_w is the water level when no ice is present, l_{well} is the length of the thermometer well within the water and r_{ice} is the radius of the ice mantle.

