

# **Final Report**

# INTER-LABORATORY COMPARISON OF TRIPLE POINT of WATER CELLS

# EURAMET. T-K7-4

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### 1. Introduction

The purpose of this comparison was to compare the results of the participating laboratories at the triple point of water temperature (TPW) and to support the Calibration and Measurement Capabilities (CMC) entries of the participating laboratories for this fixed point. This comparison was initially carried out by the support of EURAMET Focus Group.

This comparison was initiated as a EURAMET project with project number 1357. Initially, the participants of the comparison included the Metrology Institutes of Albania (DPM), F.Y.R Macedonia (BOM), Montenegro (MBM) and Serbia (DMDM). But at the later stages of the comparison, these countries understandably chose to leave the comparison instead of repeating the measurements due to an unexpected problem which is explained in section 5. On the other hand, participants from GULFMET organization, Emirates Metrology Institute (EMI) and National Measurement and Calibration Center at Saudi Standards, Metrology and Quality Organization of the Kingdom of Saudi Arabia (SASO NMCC) and also Jordan National Metrology Institute (JNMI) were included in the comparison after having approvals of each participating laboratory. Then the comparison was registered as Key Comparison with the name EURAMET.T-K7-4 in BIPM KCDB. The protocol was approved by CCT WG-KC. Finally, two loops was combined together and five institutes performed the comparison with the single circulating TPW Cell.

The objective of this comparison was to assess the uncertainty on the practical realization of triple point of water temperature by the participant laboratories.

This report presents the results of the TPW comparison and gives detailed information about the measurements made at TUBİTAK UME and participating laboratories. It is summarized the main points of the technical protocol in Chapter 3. Chapter 4 describes the transfer cell and transportation in this comparison. In Chapter 5, the problems experienced during the comparison are mentioned. Chapter 6 describes the devices used in the comparison by participants. Chapter 7 provides information on the measurements made by the participants. Chapter 8 provides a summary and the conclusions. Finally, the comparison protocol is given in the appendices.

This report contains the results reported by the participants and pilot. The pilot laboratory combined the results of the measurements in the participating laboratories and those of the cell comparison at TUBITAK UME to obtain the differences and to make the linkage to CCT K7 in this final report.

#### 2. Organization and Participants

The details of the organization of this comparison are defined in the Technical Protocol, which is reproduced in Appendix 1. Only the main points are presented in this report.

There were five participants in this intercomparison. Contact details are listed below in alphabetical order.



# Table 1. Participating Laboratories

Country (code)	Laboratory	Name of contact	Address
1) Turkey (TR)	TÜBITAK UME	Mr. Ali UYTUN Dr. Murat KALEMCİ	TÜBITAK Ulusal Metroloji Entistüsü (UME) P.O.Box 54 Gebze, Kocaeli 41470
2) Bosnia and Hercegovina (BA)	IMBIH	Miss Nedžadeta Hodžić	IMBiH, Laboratory for temperature Dolina 6 71000 Sarajevo
3) Kingdom of Saudi Arabia	SASO / NMCC	Oqab N. AlOtaibi and Dr. Ismail A. AlFaleh	National Measurement and Calibration Center at Saudi Standards, Metrology and Quality Organization of the Kingdom of Saudi Arabia, Riyadh Al Muhammadiyah –in front of King Saud University PO. B 3437 Riyadh 11471 Kingdom of Saudi Arabia
4) United Arab Emirates	EMI	Miltiadis Anagnostou	Emirates Metrology Institute, Block H, CERT Technology Park, 881, Sultan Bin Zayed The First Street, PO Box 853, Abu Dhabi, United Arab Emirates
5) Jordan ( JO)	JNMI	Eng. Mustafa Flaifel	Royal Scientific Society/Jordan National Metrology Institute, Al- Jubeiha (11941), Amman- Jordan



### 3. Protocol

The technical protocol of this comparison matches closely the corresponding CCT-K7 key comparison. The technical Protocol is reproduced in Appendix 1.

The Protocol of the EURAMET T. K-7-4 was sent to all participants and also to the CCT-WG-KC. The protocol of the comparison was finalized after making the amendments suggested by the Working Group and finally all participants have approved the protocol before the measurements began.

## 4. Description of Transfer cell and Transportation

TUBITAK UME was the pilot laboratory of the comparison and supplied the triple point of water cells to be circulated as "transfer cells" in the comparison.

Participating laboratories compared the local realization of the triple point of water and the transfer cell.

Transfer cells were manufactured by UME in 2014. Geometrical dimensions of the cells are given in Table 2.

TUBITAK UME sent transfer cell to the first participant of the comparison in a special box produced for the cell. ATA carnet was provided for the delivery of TPW cells.

	Cell	Glass	h1/mm	h2/mm	D1/mm	D2/mm
Transfer cell 1	TUBİTAK UME Zero02	Borosilicate	230	315	15	50
Transfer cell 2	TUBİTAK UME Zero03	Borosilicate	232	315	15	50

Table 2. Geometrical dimensions of transfer cell

Where,

h1: height of the water level from the bottom of the thermometer well,

h2: height of the cell body,

- d1: diameter of thermometer well,
- d2: diameter of the cell.

Upon the receipt of the transfer cell, the coordinator was informed by e-mail.

### 5. The problems experienced during the comparison

The measurements in the first loop, including EURAMET countries were completed. But unfortunately the circulating cell was broken during the transportation from last participant to TUBITAK UME. When we finished the evaluation of comparison results received from all participants of EURAMET loop, we noticed that, the temperature of TPW cell seems to shift eventually meaning that temperature deviation from the reference cell increased more and more as comparison proceeded. Finally, it was observed that the presented results by participant labs



were significantly different from the reference cell which will link our results to CCT K7 reference value. Another part of the problem was missing of final TPW measurements with this cell at UME. On the other hand no problem was observed with the transfer TPW cell of the other loop. Therefore to circulate this TPW cell in EURAMET loop was proposed to participants and asked for their approval to repeat the measurements. Only IMBIH agreed on this proposal. Hence, it was decided to perform this comparison with five participants including TUBITAK UME (Turkey) and single transfer TPW Cell. These participants are; SASO-NMCC (Kingdom of Saudi Arabia), EMI (United Arab Emirates), JNMI (Jordan) and IMBIH (Bosnia and Herzegovina).

All measurements were completed with the transfer cell (TUBİTAK UME Zero03) used in loop 2.

## 6. Devices used in the comparison

Basically, all participants used two standard platinum resistance thermometers, resistance bridge, standard resistor, triple point of water cell (national reference cell), triple point of water maintenance bath and standard resistor maintenance bath for the comparison.

All participants filled appendix A which was given in protocol (Description of Equipment used for the measurements).

Used devices in this comparison by participants are given below in the Table 3.



Table 3. Used devices by participants in this comparison

	TUBİTAK UME	EMI	SASO/ NMCC	IMBIH	JNMI
Description of national reference cell:			NWCC		
Manufacturer	TUBİTAK UME	Fluke	TUBİTAK UME	ISOTECH	Fluke
Type and Model	Wide	TPW 5901-Q	Wide	B11-50-270	5901 A-Q
Serial Number	UME 03/02	A-Q5073	12/08	B11-50-989	A-Q5055
Description of Standard Platinum Resistance Thermometer (SPRT) 1					
Manufacturer	Hart Scientific	Fluke	Hart Scientific	ISOTECH	Fluke
Model	5681	5681	5681	670 SQ /25.5	5699
Serial Number	1306	1783	1485	670 SQ/159	1118
Distance from sensor midpoint to surface level of water in transfer cell Description of Standard Platinum Resistance Thermometer (SPRT) 2	251 mm	209 mm	230 mm	197 mm	Around 204 mm
Manufacturer	Hart Scientific	Fluke	Tinsley	ACCUMAC	Isotech
Model	5681	5681	5187SA	AM1880	935-14-77E
Serial Number	1287	1787	274264	1880256	204
Distance from sensor midpoint to surface level of water in transfer cell Description of resistance bridge :	226 mm	209 mm	255 mm	209 mm	195 mm
Manufacturer	ASL	MI	ASL	ASL	Fluke
Model	F900	MI 6015T	F18	F900	8508A
Serial Number	015713/01	1102596	004879/01	015713/01	900151153
Type of resistance bridge (AC or DC): Description of standard resistor :	AC	DC	AC	AC	DC
Manufacturer	Tinsley	Tinsley	Tinsley	Tinsley	Fluke
Model	5685A	5685A	5685A/25	5685A	742A
Serial Number	269123	17894/09	6230/01	15804/23	8998009



**Table 3.** Used devices by participants in this comparison (Continuing)

	TUBİTAK UME	EMI	SASO	IMBIH	JORDAN
Description of Water triple point maintenance bath :		Water bath			
Manufacturer	ISOTECH	Fluke	ISOTECH	ISOTECH	ISOTECH
Model	18233	7312	331842-1	18233	
Serial Number	30474/1	B2B328	331842-1	30474/1	25269/6
Stability value	6 mK	0.007 °C	0.018	10 mK	0.1 mK
Description of Standard Resistor maintenance bath :		Air bath			
		Measurements			
Manufacturer	Fluke	International	Fluke	ISOTECH	None
Model	7008	MI-9300A	7008	455	None
Serial Number	B02332	1103442	B431	30474/9	None
Stability value	5 mK	23 °C ±0.02 °C	0.001 °C	12mK	None
Measurement current:	1mA and √2mA	1 mA	1mA, √2mA, 1mA	1mA and √2mA	1 mA, 1.4 mA
Number and sampling frequency of repeated measurements:	30	36, 8 s reversal time	30 data, 10 s	30	None
			SPRT1: Pt-25	SPRT1: 25	
		Fluke 5681	Ohm, 50 mm	Ohm,40mm;	
Time of the sum of the	Hart Scientific, SPRT 25 ohm,	25.5 ohm, 30	SPRT2: Pt-25	SPRT2 :25	
Type of thermometer, length of sensor:	48 mm	mm	Ohm, 45 mm	Ohm, 42mm	None

#### 7. Measurements and Uncertainty

The temperature realized in the transfer TPW cell was compared with the one obtained with the national reference cell of participant laboratory using standard platinum resistance thermometers (SPRTs) according to this protocol.

Transfer and reference triple point of water cells were placed in the maintenance bath for a period of 10 day prior to the measurements. The transfer TPW cell was compared with the reference TPW cell, at least for a total of 10 days using two different SPRTs (one cycle). Each participant performed at least 2 cycles of measurement including a new realization of the ice mantle.

Measurements were performed at 1 mA and  $\sqrt{2}$  mA for extrapolation for 0 mA value.

The triple point of water (TPW) temperature values given in this comparison result were the values obtained after applying hydrostatic head and self-heating corrections for the measured values.



The comparison was performed by measuring the difference in temperature between the transfer and national reference TPW cells. The difference in the observed resistances (corrected for the hydrostatic head effect and self-heating, and possible calibration of the measurement instrument) for the two cells was converted to a temperature difference using the dT/dR for the SPRT's.

T (national cell) - T (transfer cell) =(R (national cell) - R (transfer cell)) × dT/dR (6.1)

In this comparison, the measurement results made by the participants are given in Table 4 to 8.

The uncertainty was assessed based on the results given in Table 4 to 8 and according to Table 4 given in the technical protocol. Also, measurement results for immersion profile by the participants are given in Table 9 to 13.

All participants filled in the uncertainty budget as given in appendix D. The uncertainty budgets filled by participants of this comparison are given in Table 14 to 18.



# Table 4.1. Measurement results on first ice mantle at TUBİTAK UME before intercomparison

	Date of preparation of ice mantle of transfer cell:02.01.2015Date of preparation of the mantle of the reference cell:02.01.2015											
Meas.No.	Date of		SPRT 1			SPRT 2						
	measurement	Difference (T(transfer)- T(nat. ref.)	Experimental standard deviation of the mean of T(transfer)	Experimental standard deviation of the mean of T(nat. ref.)	Difference (T(transfer)- T(nat. ref.)	Experimental standard deviation of the mean of T(transfer)	Experimental standard deviation of the mean of T(nat. ref.)					
	Unit		K	101.7		K	101.)					
1	12.01.2015	-0.000090	0.000004	0.000006	-0.000027	0.000003	0.000005					
2	13.01.2015	-0.000020	0.000004	0.000005	-0.000018	0.000006	0.000005					
3	14.01.2015	-0.000044	0.000004	0.000005	-0.000064	0.000005	0.000006					
4	15.01.2015	-0.000056	0.000006	0.000005	-0.000085	0.000005	0.000004					
5	16.01.2015	-0.000028	0.000006	0.000006	-0.000015	0.000007	0.000005					
6	17.01.2015	-0.000035	0.000004	0.000004	0.000057	0.000007	0.000004					
7	18.01.2015	-0.000035	0.000005	0.000004	-0.000039	0.000005	0.000005					
8	19.01.2015	-0.000024	0.000005	0.000003	-0.000054	0.000004	0.000005					
9	20.01.2015	-0.000081	0.000005	0.000007	-0.000071	0.00006	0.000005					
10	21.01.2015	-0.000080	0.000006	0.000007	-0.000026	0.000004	0.000007					
		К	mK		K	mK						
Av	verage:	-0.000049	-0.0494		-0.000034	-0.0342						



#### Table 4.2. Measurement results on second ice mantle at before intercomparison

Date of preparation of ice mantle of transfer cell: 04.05.2015

Date of preparation of the mantle of the reference cell: 04.05.2015

Meas.No.	Date of		SPRT 1			SPRT 2	
	measurement	Difference	Experimental standard	Experimental	Difference	Experimental standard	Experimental standard
		(T(transfer)-	deviation of the mean	standard deviation of	(T(transfer)-	deviation of the mean of	deviation of the mean
		T(nat. ref.)	of T(transfer)	the mean of T(nat.	T(nat. ref.)	T(transfer)	of T(nat. ref.)
				ref.)			
	Unit		K			K	
1	15.05.2015	-0.000056	0.000004	0.000006	-0.000014	0.000004	0.000004
2	16.05.2015	0.000024	0.000003	0.000006	-0.000022	0.000004	0.000004
3	17.05.2015	-0.000120	0.000006	0.000004	-0.000038	0.000005	0.000006
4	18.05.2015	0.000021	0.000008	0.000005	-0.000045	0.000004	0.000005
5	19.05.2015	-0.000119	0.000005	0.000006	-0.000093	0.00006	0.000005
6	20.05.2015	0.000104	0.000005	0.000005	-0.000061	0.000004	0.000006
7	21.05.2015	0.000077	0.000005	0.000004	-0.000054	0.00006	0.000004
8	22.05.2015	-0.000132	0.000006	0.000005	-0.000123	0.000005	0.000004
9	23.05.2015	-0.000046	0.000004	0.000004	0.000065	0.000005	0.000006
10	24.05.2015	0.000035	0.000005	0.000004	0.000088	0.000006	0.000004
		K	mK		K	mK	
Av	verage:	-0.000021	-0.021		-0.000030	-0.0296	

The average difference between the transfer cell and the national reference obtained with both SPRTS was -0.034 mK for TUBITAK UME before the comparison.



#### Table 4.3. Measurement results on first ice mantle at TUBİTAK UME end of intercomparison

Date of preparation of ice mantle of transfer cell:20.02.2019Date of preparation of the mantle of the reference cell:20.02.2019

Meas.No.	Date of		SPRT 1			SPRT 2	
	measurement	Difference	Experimental	Experimental	Difference	Experimental standard	Experimental
		(T(transfer)-	standard deviation of	standard deviation of	(T(transfer)-	deviation of the mean of	standard deviation of
		T(nat. ref.)	the mean of	the mean of T(nat.	T(nat. ref.)	T(transfer)	the mean of T(nat.
			T(transfer)	ref.)			ref.)
	Unit		K			K	
1	02.03.2019	-0.000039	0.000005	0.000009	-0.000074	0.000003	0.000006
2	03.03.2019	-0.000015	0.000008	0.000007	-0.000057	0.000003	0.000004
3	04.03.2019	-0.000019	0.000006	0.000006	-0.000018	0.000006	0.000004
4	05.03.2019	0.000057	0.000006	0.000005	0.000043	0.000006	0.000006
5	06.03.2019	0.000029	0.000005	0.000004	-0.000079	0.000006	0.000005
6	08.03.2019	-0.000013	0.000006	0.000004	0.00008	0.000004	0.000005
7	09.03.2019	0.000047	0.000005	0.000005	-0.000074	0.000005	0.000005
8	10.03.2019	-0.000051	0.000005	0.000003	-0.000097	0.000004	0.000003
9	11.03.2019	0.000069	0.000005	0.000006	0.000004	0.000006	0.000009
10	12.03.2019	-0.000083	0.000006	0.000007	-0.000088	0.000005	0.000007
		К	mK		К	mK	
Av	erage:	-0,000002	-0,0021		-0.000043	-0.0431	



#### Table 4.4. Measurement results on second ice mantle at end of intercomparison

Date of preparation of ice mantle of transfer cell: 15.03.2019

Date of preparation of the mantle of the reference cell: 15.03.2019

Meas.No.	Date of		SPRT 1			SPRT 2	
	measurement	Difference	Experimental standard	Experimental	Difference	Experimental standard	Experimental standard
		(T(transfer)-	deviation of the mean	standard deviation of	(T(transfer)-	deviation of the mean of	deviation of the mean
		T(nat. ref.)	of T(transfer)	the mean of T(nat.	T(nat. ref.)	T(transfer)	of T(nat. ref.)
				ref.)			
	Unit		K			K	
1	25.03.2019	-0.000008	0.000009	0.000006	-0.000014	0.00008	0.00005
2	26.03.2019	0.000084	0.000005	0.000006	-0.000022	0.000007	0.00007
3	27.03.2019	-0.000102	0.000007	0.000004	-0.000038	0.000006	0.00006
4	28.03.2019	-0.000030	0.000006	0.000003	-0.000045	0.000006	0.00005
5	29.03.2019	-0.000018	0.000004	0.000004	-0.000093	0.000005	0.00005
6	30.03.2019	-0.000004	0.000005	0.000005	-0.000061	0.00006	0.00003
7	31.03.2019	-0.000042	0.000005	0.000007	-0.000054	0.00006	0.00007
8	01.04.2019	0.000011	0.000004	0.000005	-0.000123	0.000004	0.00005
9	02.04.2019	-0.000036	0.000004	0.000004	0.000082	0.000004	0.000004
10	03.04.2019	0.000024	0.000005	0.000005	-0.000046	0.000006	0.00003
		ĸ	mK		K	mK	
Av	erage:	-0.000012	-0.0121		-0.000041	-0.0414	

The average difference between the transfer cell and the national reference obtained with both SPRTS was -25 µK for TUBITAK UME at the end of the comparison.

The average difference obtained before and after the comparison with both SPRTS was calculated as -29.5 µK.



#### Table 5.1. Measurement results on first ice mantle at EMI

Date of preparation of ice mantle of transfer cell: 17.11.2016

Date of preparation of the mantle of the reference cell: 17.11.2016

Meas.No.	Date of		SPRT 1			SPRT 2	
	measurement	Difference	Experimental	Experimental	Difference	Experimental standard	Experimental
		(T(transfer)-	standard deviation of	standard deviation of	(T(transfer)-	deviation of the mean of	standard deviation of
		T(nat. ref.)	the mean of	the mean of T(nat.	T(nat. ref.)	T(transfer)	the mean of T(nat.
			T(transfer)	ref.)			ref.)
	Unit		K			К	
1	04.12.2016	-0.000112	0.000006	0.000006	-0.000097	0.000007	0.00008
2	05.12.2016	-0.000099	0.000006	0.000006	-0.000104	0.000005	0.000006
3	06.12.2016	-0.000107	0.000010	0.000006	-0.000089	0.000005	0.000004
4	07.12.2016	-0.000091	0.000004	0.000007	-0.000077	0.000005	0.000005
5	08.12.2016	-0.000069	0.000005	0.000006	-0.000093	0.000004	0.000009
6	12.12.2016	-0.000097	0.000004	0.000006	-0.000098	0.000005	0.000005
7	14.12.2016	-0.000116	0.000005	0.000006	-0.000079	0.000006	0.000005
8	15.12.2016	-0.000090	0.000004	0.000004	-0.000101	0.000005	0.00006
9	18.12.2016	-0.000110	0.000005	0.000004	-0.000089	0.000005	0.000006
10	18.12.2016	-0.000080	0.000004	0.000003	-0.000099	0.000005	0.000004
		К	mK		К	mK	
Av	erage:	-0.000097	-0.097		-0.000093	-0.093	



#### Table 5.2. Measurement results on second ice mantle at EMI

Date of preparation of ice mantle of transfer cell: 22.12.2016

Date of preparation of the mantle of the reference cell: 22.12.2016

Meas.No.	Date of		SPRT 1		SPRT 2			
	measurement	Difference (T(transfer)- T(nat. ref.)	Experimental standard deviation of the mean of T(transfer)	Experimental standard deviation of the mean of T(nat. ref.)	Difference (T(transfer)- T(nat. ref.)	Experimental standard deviation of the mean of T(transfer)	Experimental standard deviation of the mean of T(nat. ref.)	
	Unit		К			К		
1	02.01.2017	-0.000080	0.000004	0.000005	-0.000105	0.000005	0.000007	
2	03.01.2017	-0.000084	0.000005	0.000009	-0.000072	0.00006	0.000006	
3	10.01.2017	-0.000086	0.000005	0.000004	-0.000085	0.00006	0.000005	
4	11.01.2017	-0.000073	0.000005	0.000002	-0.000098	0.000005	0.000004	
5	12.01.2017	-0.000118	0.000007	0.000004	-0.000086	0.00006	0.000003	
6	15.01.2017	-0.000106	0.000008	0.000004	-0.000111	0.000005	0.000005	
7	17.01.2017	-0.000090	0.000006	0.000003	-0.000094	0.000005	0.000005	
8	18.01.2017	-0.000084	0.000005	0.000004	-0.000102	0.00006	0.000004	
9	18.01.2017	-0.000060	0.000004	0.000004	-0.000071	0.000005	0.00007	
10	19.01.2017	-0.000085	0.000003	0.000004	-0.000092	0.000005	0.000005	
		K	mK		K	mK		
Av	erage:	-0.000087	-0.088		-0.000092	-0.092		

The average difference between the transfer cell and the national reference obtained with both SPRTS was -92.0 µK for EMI at the comparison.



#### Table 6.1. Measurement results on first ice mantle at SASO/NMCC

Date of preparation of ice mantle of transfer cell: Date of preparation of the mantle of the reference cell:

04.03.2018 04.03.2018

Meas.No.	Date of		SPRT 1			SF	PRT 2
	measurement	Difference (T(transfer)- T(nat. ref.)	Experimental standard deviation of the mean of T(transfer)	Experimental standard deviation of the mean of T(nat. ref.)	Difference (T(transfer) -T(nat. ref.)	Experimental standard deviation of the mean of T(transfer)	Experimental standard deviation of the mean of T(nat. ref.)
	Unit		Ohm			1 /	Dhm
1	14.03.2018	0.000 008 2	0.000 004 7	0.000 004 6	0.000 006 8	0.000 001 7	0.000 001 5
2	15.03.2018	0.000 016 7	0.000 002 9	0.000 003 2	0.000 014 4	0.000 002 8	0.000 002 6
3	16.03.2018	0.000 017 0	0.000 003 4	0.000 001 9	0.000 015 6	0.000 003 3	0.000 001 8
4	17.03.2018	0.000 014 5	0.000 002 2	0.000 002 3	0.000 010 6	0.000 002 8	0.000 004 3
5	18.03.2018	0.000 014 1	0.000 004 5	0.000 002 7	0.000 018 7	0.000 002 1	0.000 002 3
6	19.03.2018	0.000 017 6	0.000 003 2	0.000 004 8	0.000 016 4	0.000 003 4	0.000 002 3
7	20.03.2018	0.000 016 9	0.000 004 0	0.000 004 1	0.000 015 0	0.000 001 9	0.000 003 0
8	21.03.2018	0.000 013 7	0.000 002 3	0.000 002 9	0.000 015 2	0.000 003 4	0.000 003 1
9	22.03.2018	0.000 018 2	0.000 001 4	0.000 002 2	0.000 015 7	0.000 002 2	0.000 003 1
10	23.03.2018	0.000 020 9	0.000 002 6	0.000 002 3	0.000 015 9	0.000 003 8	0.000 002 4
			mK			mK	
Av	erage:	0.0000158	0.158		0.0000144	0.144	



#### Table 6.2. Measurement results on second ice mantle at SASO/NMCC

Date of preparation of ice mantle of transfer cell:25.0Date of preparation of the mantle of the reference cell:25.0

25.03.2018 25.03.2018

Meas.No.	Date of	SPRT 1			SPRT 2		
	measurement	Difference	Experimental standard	Experimental standard	Difference	Experimental	Experimental standard
		(T(transfer)-	deviation of the mean	deviation of the mean of	(T(transfer)-	standard	deviation of the mean of T(nat.
		T(nat. ref.)	of T(transfer)	T(nat. ref.)	T(nat. ref.)	deviation of the	ref.)
						mean of T(transfer)	
	Unit		Ohm			Oh	m
1	04.04.2018	0.000 004 1	0.000 003 9	0.000 003 3	0.000 008 0	0.000 002 8	0.000 002 3
2	05.04.2018	0.000 000 3	0.000 002 3	0.000 003 5	0.000 004 8	0.000 003 1	0.000 000 0
3	06.04.2018	0.000 006 0	0.000 000 8	0.000 001 4	0.000 009 8	0.000 000 6	0.000 001 2
4	07.04.2018	0.000 012 3	0.000 000 9	0.000 001 6	0.000 013 7	0.000 001 3	0.000 001 5
5	08.04.2018	0.000 007 4	0.000 001 1	0.000 000 6	0.000 013 8	0.000 001 1	0.000 001 4
6	09.04.2018	0.000 008 6	0.000 001 0	0.000 000 9	0.000 012 0	0.000 001 4	0.000 001 1
7	10.04.2018	0.000 014 2	0.000 001 7	0.000 001 1	0.000 007 6	0.000 001 2	0.000 001 6
8	11.04.2018	0.000 010 9	0.000 001 1	0.000 001 2	0.000 016 8	0.000 001 4	0.000 001 5
9	12.04.2018	0.000 013 0	0.000 001 2	0.000 001 9	0.000 010 9	0.000 001 3	0.000 001 1
10	13.04.2018	0.000 011 8	0.000 001 0	0.000 001 5	0.000 011 8	0.000 001 3	0.000 001 4
			mK			mK	
Av	verage:	0.000089	0.089		0.0000106	0.106	

The average difference between the transfer cell and the national reference obtained with both SPRTS was 124.0 µK for SASO at the comparison.



#### Table 7.1. Measurement results on first ice mantle at IMBIH

Date of preparation of ice mantle of transfer cell:02.11.2018Date of preparation of the mantle of the reference cell:02.11.2018

Meas.No.	Date of		SPRT 1		SPRT 2		
	measurement	Difference	Experimental	Experimental standard	Difference	Experimental	Experimental standard deviation
		(T(transfer)-	standard deviation of	deviation of the mean of	(T(transfer)	standard	of the mean of T(nat. ref.)
		T(nat. ref.)	the mean of	T(nat. ref.)	-T(nat. ref.)	deviation of the	
			T(transfer)			mean of T(transfer)	
	Unit		К				К
1	12.11.18	0.00000153	7.237E-05	5.620E-05	0.0000533	5.738E-05	7.237E-05
2	13.11.18	0.00008699	4.955E-05	4.110E-05	0.0000108	7.839E-05	6.544E-05
3	14.11.18	-0.00013901	5.135E-05	4.735E-05	-0.0000399	6.929E-05	6.404E-05
4	15.11.18	-0.00002774	4.468E-05	4.468E-05	-0.0000277	5.659E-05	5.659E-05
5	16.11.19	0.00001713	5.602E-05	3.785E-05	0.0000165	6.111E-05	6.637E-05
6	19.11.19	-0.00010641	4.047E-05	4.957E-05	0.0000268	5.658E-05	5.842E-05
7	20.11.19	-0.00001987	5.663E-05	6.031E-05	-0.0001132	7.033E-05	6.734E-05
8	21.11.19	-0.00002481	4.971E-05	4.337E-05	-0.0000411	7.345E-05	7.612E-05
9	22.11.19	-0.00002687	6.925E-05	5.058E-05	0.0000188	9.053E-05	6.334E-05
10	23.11.19	0.00002479	4.054E-05	4.807E-05	-0.0003062	6.218E-05	6.029E-05
			mK			mK	
Av	verage:	-0.000021	-0.021		-0.000040	-0.040	



#### Table 7.2. Measurement results on second ice mantle at IMBIH

Date of preparation of ice mantle of transfer cell:03.1Date of preparation of the mantle of the reference cell:03.1

03.12.2018 03.12.2018

Meas.No.	Date of		SPRT 1			SPRT 2	
	measurement	Difference	Experimental standard	Experimental standard	Difference	Experimental	Experimental
		(T(transfer)-	deviation of the mean	deviation of the mean	(T(transfer)-	standard	standard deviation of
		T(nat. ref.)	of T(transfer)	of T(nat. ref.)	T(nat. ref.)	deviation of the mean of	the mean of T(nat. ref.)
						T(transfer)	1ei.)
	Unit		К			К	
1	13.12.2018	-0.00004907	5.649E-05	6.401E-05	-0.00001327	5.858E-05	4.463E-05
2	14.12.2018	0.00014776	6.235E-05	7.770E-05	0.00000566	5.817E-05	6.418E-05
3	17.12.2018	0.00006756	7.538E-05	6.694E-05	-0.00018341	4.471E-05	5.554E-05
4	18.12.2018	0.00013416	7.409E-05	4.929E-05	-0.00010421	5.895E-05	5.268E-05
5	19.12.2019	0.00001209	7.526E-05	7.761E-05	-0.00009847	4.790E-05	5.345E-05
6	20.12.2018	-0.00011787	6.817E-05	9.902E-05	-0.00009361	5.139E-05	4.920E-05
7	21.12.2018	-0.00003024	7.501E-05	5.437E-05	-0.00011814	5.414E-05	4.152E-05
8	24.12.2018	0.00005753	5.868E-05	6.623E-05	-0.00002057	7.335E-05	7.156E-05
9	25.12.2018	-0.00014787	5.991E-05	4.709E-05	-0.00009047	4.763E-05	8.302E-05
10	26.12.2018	-0.00000647	6.957E-05	7.269E-05	0.00010359	4.969E-05	5.615E-05
			mK			mK	
Av	erage:	0.000007	0.007		-0.000006	-0.061	

The average difference between the transfer cell and the national reference obtained with both SPRTS was -29.0 µK for IMBIH at the comparison.



#### Table 8.1. Measurement results on first ice mantle at JNMI

Date of preparation of ice mantle of transfer cell: Date of preparation of the mantle of the reference cell:

Meas.No.	Date of		SPRT 1	SPRT 1		SPRT 2	
	measurement	Difference (T(transfer)- T(nat. ref.)	Experimental standard deviation of the mean of T(transfer)	Experimental standard deviation of the mean of T(nat. ref.)	Difference (T(transfer)- T(nat. ref.)	Experimental standard deviation of the mean of T(transfer)	Experimental standard deviation of the mean of T(nat. ref.)
	Unit		K			K	
1	14.03.2018	0.0002	1.000E-04	1.000E-04	0.0001	1.000E-04	1.000E-04
2	15.03.2018	0.0002	1.000E-04	1.000E-04	0.0001	1.000E-04	1.000E-04
3	16.03.2018	0.0002	1.000E-04	1.000E-04	0.0001	1.000E-04	1.000E-04
4	17.03.2018	0.0002	1.000E-04	1.000E-04	0.0001	1.000E-04	1.000E-04
5	18.03.2018	0.0002	1.000E-04	1.000E-04	0.0001	1.000E-04	1.000E-04
6	19.03.2018	0.0002	1.000E-04	1.000E-04	0.0001	1.000E-04	1.000E-04
7	20.03.2018	0.0002	1.000E-04	1.000E-04	0.0001	1.000E-04	1.000E-04
8	21.03.2018	0.0002	1.000E-04	1.000E-04	0.0001	1.000E-04	1.000E-04
9	22.03.2018	0.0002	1.000E-04	1.000E-04	0.0001	1.000E-04	1.000E-04
10	23.03.2018	0.0002	1.000E-04	1.000E-04	0.0001	1.000E-04	1.000E-04
			mK			mK	
Av	erage:	0.0002	0.2		0.0001	0.1	

The average difference between the transfer cell and the national reference obtained with both SPRTS was 150 µK for JNMI at the comparison. It should be mentioned that JNMI carried out only one set of measurements unlike the other participants of the comparison.



### Table 9. Measurement results for immersion profile obtained by UME

Immersion profile (for transfer cell)

Distance from sensor midpoint to free surface level of the liquid water (mm)	Temperature variation (mK)	Expected variation (mK)
210	0.000	0.000
200	-0.043	0.007
190	-0.039	0.015
180	0.004	0.022
170	0.003	0.029
160	0.066	0.037
150	0.016	0.044

Immersion profile (for national reference cell)

Distance from sensor midpoint to free surface level of the liquid water (mm)	Temperature variation (mK)	Expected variation (mK)
250	0.000	0.000
240	0.049	0.007
230	-0.004	0.015
220	0.057	0.022
210	0.045	0.029
200	0.039	0.037
190	0.040	0.044

#### Table 10. Measurement results for immersion profile obtained by EMI

Immersion profile (for transfer cell)

Distance from sensor midpoint to free surface level of the liquid water (cm)	Temperature variation (mK)	Expected variation (mK)
20.9	0.000	0.000
19.9	-0.006	0.007
18.9	0.033	0.015
17.9	0.022	0.022
16.9	0.020	0.029
15.9	0.056	0.037
14.9	0.022	0.044

Immersion profile (for national reference cell)

	/	
Distance from sensor midpoint to free surface level of the liquid water (cm)	Temperature variation (mK)	Expected variation (mK)
25.9	0.000	0.000
24.9	0.019	0.007
23.9	0.038	0.015
22.9	0.044	0.022
21.9	0.045	0.029
20.9	0.046	0.037
19.9	0.051	0.044



# **UME** Table 11. Measurement results for immersion profile obtained by SASO NMCC

Immersion profile (for national refere	ence cell) UME 12/08 (SASO)	Immersion profile (for transfer cell) UME	E Zero 3 (UME)
Distance from sensor midpoint to free surface level of the liquid water (mm)	Temperature variation (mK)	Distance from sensor midpoint to free surface level of the liquid water (mm)	Temperature variation (mK)
255	0.000	230	0.000
245	-0.053	220	-0.024
235	-0.026	210	0.009
225	-0.020	200	-0.005
215	-0.033	190	-0.005
205	0.041	180	-0.015
195	-0.021	170	-0.023

# Table 12. Measurement results for immersion profile obtained by IMBIH

(with Isotech SPRT)	Transfer Cell
Distance from sensor	Temperature variation
midpoint to free surface level	(K)
of the liquid water	
0 (19,7)	0.0000000
1 (18,7)	0.0000870
2 (17,7)	-0.0000177
3 (16,7)	0.0001634
4 (15,7)	0.0001756
5 (14,7)	0.0001376
6 (13,7)	0.0000110

(with Isotech SPRT)	National Cell
Distance from sensor midpoint to	Temperature variation
free surface level of the liquid	(K)
water	
0 (23,5)	0.0000000
1 (22,5)	-0.0000982
2 (21,5)	0.0000697
3 (20,5)	0.0000288
4 (19,5)	0.0001408
5 (18,5)	-0.0000010
6 (17,5)	-0.000072



# Table 13. Measurement results for immersion profile obtained by JNMI

Immersion profile (for transfer cell)			
Distance from sensor midpoint	Temperature variation		
to free surface level of the liquid			
water			
20 cm	0.0 mK		
19 cm	0.0 mK		
18 cm	0.0 mK		
17 cm	0.0 mK		
16 cm	0.0 mK		
15 cm	0.0 mK		
14 cm	0.1 mK		

Immersion profile (for national reference cell)

Distance from sensor	Temperature	
midpoint to free surface	variation	
level of the liquid water		
22 cm	0.0 mK	
21 cm	0.0 mK	
20 cm	0.0 mK	
19 cm	0.0 mK	
18 cm	0.0 mK	
17 cm	0.0 mK	
16 cm	0.0 mK	



# Table 14. Triple Point of Water Comparison Uncertainty Budget for UME

Quantity	Component Description	Standard uncertainty contribution (k=1)
Qi		u <sub>i</sub> in mK
1	Chemical impurities	0.0202
2	Isotopic variation	0.0300
3	Repeatability for a single ice mantel (incl. bridge noise)	0.0110
4	Reproducibility for different ice mantles	0.0020
5	Reproducibility different types of SPRT	0.0007
6	Hydrostatic head of transfer cell	0.0073
7	Hydrostatic head of national reference cell	0.0073
8	Perturbing heat exchanges	0.0200
9	SPRT self-heating in the transfer cell and reference cell	0.0270
10	others	
Combined	uncertainty	0.051
Expanded u	uncertainty ( k = 2)	0.102



## Table 15. Triple Point of Water Comparison Uncertainty Budget for EMI

Quantity		Standard uncertainty contribution (k=1)
	National reference (Uncertainties related	in mK
Qi	only to properties of the reference cell)	
1	Chemical impurities	0.029
2	Isotopic variation	0.029
3	Residual gas pressure in cell	0.005
4	Reproducibility	0.030
	Comparison of transfer cell to national reference (Uncertainties related to the comparison of the two cells)	
5	Repeatability for a single ice mantel (incl. bridge noise)	0.005
6	Reproducibility for different ice mantles	0.006
7	Reproducibility for different types of SPRTs	0.003
8	Hydrostatic head of transfer cell	0.007
9	Hydrostatic head of national reference cell	0.007
10	Perturbing heat exchanges	0.014
11	SPRT self-heating in the transfer cell and reference cell	0.023
	others	
	Combined standard uncertainty	0.06
	Expanded uncertainty ( k = 2)	0.118

## Table 16. Triple Point of Water Comparison Uncertainty Budget for SASO

Quantity	Component Description	Standard uncertainty contribution (k=1)
Qi		u <sub>i</sub> in mK
1	Chemical impurities	0.07
2	Isotopic variation	0.001
3	Repeatability for a single ice mantel (incl. bridge noise)	0.04
4	Reproducibility for different ice mantles and different SPRT	0.03
5	Hydrostatic head of transfer cell	0.01
6	Hydrostatic head of national reference cell	0.01
7	Perturbing heat exchanges	0.15
8	SPRT self-heating in the transfer cell and reference cell	0.09
9	others	0.05
Combined u	uncertainty	0.20
Expanded u	uncertainty ( k = 2)	0.40



## Table 17. Triple Point of Water Comparison Uncertainty Budget for IMBIH

Quantity	Component Description	Standard uncertainty contribution (k=1)
Qi		u <sub>i</sub> in mK
1	Chemical impurities	0.020
2	Isotopic variation	0.020
3	Repeatability for a single ice mantel (incl. bridge noise)	0.099
4	Reproducibility for different ice mantles and different SPRT	0.020
5	Hydrostatic head of transfer cell	0.015
6	Hydrostatic head of national reference cell	0.015
7	Perturbing heat exchanges	0.018
8	SPRT self-heating in the transfer cell and reference cell	0.120
9	others	-
Combined	uncertainty	0.1618
Expanded	uncertainty ( k = 2)	0.324

Table 18. Triple Point of Water Comparison Uncertainty Budget for JNMI

Quantity	Component Description	Standard uncertainty contribution (k=1)
Qi		u <sub>i</sub> in mK
1	Chemical impurities	0.012
2	Isotopic variation	0.004
3	Repeatability for a single ice mantel (incl. bridge noise)	0.058
4	Reproducibility for different ice mantles and different SPRT	0.058
5	Hydrostatic head of transfer cell	0.058
6	Hydrostatic head of national reference cell	0.058
7	Perturbing heat exchanges	0.058
8	SPRT self-heating in the transfer cell and reference cell	0.23
9	Measuring System	0.07
Combined	uncertainty	0.48
Expanded	uncertainty ( k = 2)	0.96



### 8. Comparison of the National References

#### • Differences between the national references and the UME reference

The deviations of the national reference cells from the TUBİTAK UME reference are obtained from the results of the comparison of the transfer cells at the TUBİTAK UME and from the calibration results provided by the laboratories.

$$\Delta T_{lab,i} = T_{lab,i} - T_{UME} = \left(T_{transfer,i} - T_{UME}\right) - \left(T_{transfer,i} - T_{lab,i}\right)$$
(8.1)

Where T<sub>ref,i</sub> and T<sub>transfer,i</sub> are the temperatures of the reference cell(s) and the transfer cell of

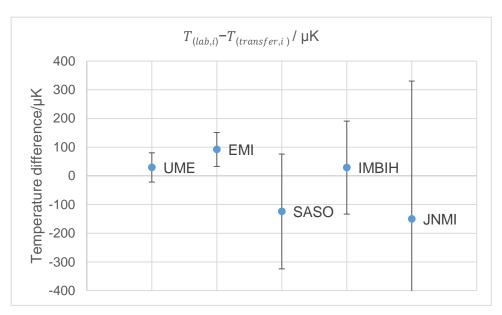
Laboratory i and  $T_{ref,UME}$  is the temperature attributed to the TUBİTAK UME.

The results are shown in Figure 1, the numerical values are given in Table 19.

# **Table 19.** Temperature differences between the national references and the UME reference.

Laboratory	$T_{lab,i} - T_{transfer,i}$ / $\mu K$	$T_{lab,i} - T_{UME}$ / $\mu K$	u <sub>lab</sub> / µK (k=1)
UME	29.5	0.0	51
EMI	92.0	62.5	59
SASO	-124.0	-153.5	200
IMBIH	29.0	-0.5	162
JNMI	-150.0	-179.5	480





**Figure 1**: Differences of the transfer cell from the national reference cells of Labs. The standard uncertainty includes the uncertainty of the comparison at the UME and the calibration uncertainty stated by the participants, including the realization uncertainty of the TPW.



## 9. Linkage to the CCT Comparisons (Linking Measurement Results to the KCRV)

The linkage to the CCT comparison has been made from the differences obtained between UME and EMI, SASO, IMBIH and JNMI and the difference that UME obtained in the CCT-K7 comparison.

The linkage to the mother CCT comparison (CCT-K7) was accomplished through the steps mentioned below:

The Pilot Laboratory (PL) calculated the  $KCRV_{LINK}$  by making use of PL Degrees of Equivalence (DoE) with the CCT K-7 KCRV, as shown in Eq. 9.1.

$$T_{UME} - T_{KCRV} = -75 \,\mu K \,(CCT \,K - 7 \,final \,report \,Table 21, page 70.)$$
(9.1)

However, no correction for isotopic composition was applied to UME results as mentioned in the page 60 of Final Report of CCT K7. Later, the studies on the isotopic composition of UME cells were carried out and published (*"Recent Developments in TUBITAK UME-made Triple Point of Water Cells", International Metrology Conference CAFMET 2008*). The isotopic correction for UME TPW cell was calculated as 47 µK.

Therefore considering the results shown in Table 19 and the correction due to isotopic composition, the new  $T_{UME} - T_{KCRV}$  would be,

$$\Delta T_{LINK} = T_{UME} - T_{KCRV} = -75 + (47) \ \mu K = -28.0 \ \mu K \tag{9.2}$$

The uncertainty of  $\Delta T_{LINK}$  (the deviation of the UME from the KCRV) at k=1 is calculated as in Eq. 9.3. :

$$u(\Delta T_{LINK})^2 = u_{\Delta T1}^2 + u(\Delta T_{PL_{CCTK7}})^2$$
(9.3)

 $u(\Delta T_{LINK}) = 96.2 \,\mu\text{K}$ 

Finally, the ( $T_{lab,i} - T_{UME}$ ) results given in Table 19 and  $\Delta T_{LINK}$  values are employed to establish the link to KCRV as shown below and the final results are presented in Table 20.

Deviation of each NMI values (*di*) from the *KCRV*<sub>LINK</sub> is calculated as in Eq. 9.4:

$$d_i = \Delta T_i + \Delta T_{LINK} \tag{9.4}$$

The uncertainty of data 95% level of confidence is calculated as in Eq. 9.5 and 9.6:

$$u_d^2 = u(\Delta T_{LINK})^2 + u_i^2$$
(9.5)

$$U_d = k \cdot u_d, \text{ where } k = 2 \tag{9.6}$$

The comparison results relative to the KCRV are shown in Figure 2 and are listed in Table 20.



Table 20. The temperature deviations of NMIs from  $KCRV_{LINK}$  and the associated standard uncertainties

Laboratory	Deviation from <i>KCRV<sub>LINK</sub></i> / μK	Std. unc. of Deviation / μΚ
UME	-28.0	96.2
EMI	34.5	113.7
SASO	-181.5	220.6
IMBIH	-28.5	188.9
JNMI	-207.5	489.7

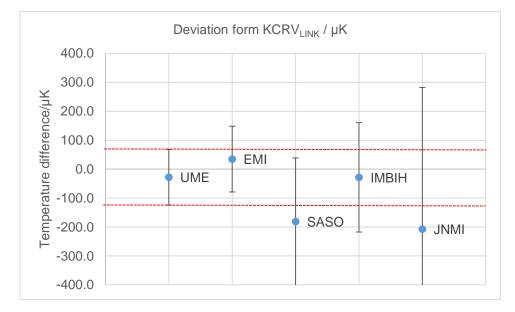


Figure 3: The comparison results relative to the KCRV. All uncertainty bars are calculated for k=1.



#### 10. Summary

As mentioned earlier, this comparison was initiated as a EURAMET project with project number 1357. Initially, the participants of the comparison included the Metrology Institutes of Albania (DPM), F.Y.R Macedonia (BOM), Montenegro (MBM) and Serbia (DMDM). But at the later stages of the comparison, these countries chose to leave the comparison instead of repeating the measurements due to an unexpected problem which is explained previously. On the other hand, participants from GULFMET organization, Emirates Metrology Institute (EMI) and National Measurement and Calibration Center at Saudi Standards, Metrology and Quality Organization of the Kingdom of Saudi Arabia (SASO NMCC) and also Jordan National Metrology Institute (JNMI) were included in the comparison after having approvals of each participating laboratory. Then the comparison was registered as Key Comparison with the name EURAMET.T-K7-4 in BIPM KCDB. The protocol was also approved by CCT WG-KC. Finally, five institutes performed the comparison with the single circulating TPW Cell.

The results sent by the participants are given in this report. Besides, the linkage to CCT-K7 KCRV was established through the deviations from the circulating cell and then through the difference between the circulating cell and the UME National Reference TPW temperature.

Finally, it can be concluded that the comparison was successful since each participant was able to cover the reference value within the limits of their given uncertainty.

#### 11. References

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Appendix A Inter-Laboratory Comparison of Triple Point of Water Cells Technical Protocol



# TUBITAK UME TEMPERATURE LABORATORY

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# INTER-LABORATORY COMPARISON OF TRIPLE POINT of WATER CELLS TECHNICAL PROTOCOL

FINAL

Agreed EURAMET Project N° 1357

(UME-G1SI-15-01)

# **Coordinators:**

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### 1. Introduction

The purpose of this comparison is to compare the results of the participating laboratories during comparison at the triple point of water and to support the Calibration and Measurement Capabilities (CMC) entries of the participating laboratories.

This protocol describes the objectives of the comparison, its organization and the procedures to be followed by the participants during the measurements and evaluation. This comparison is carried out by the support of EURAMET Focus Group .

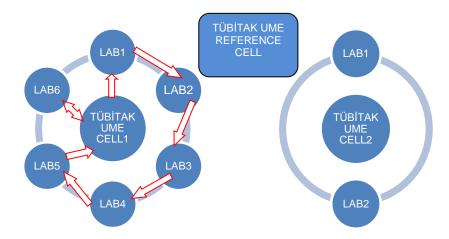
The objective of this comparison is to assess the uncertainty of the practical realization of triple point of water by the participant laboratories.

TUBITAK UME is the pilot laboratory of the comparison and will supply the triple point of water cell to be circulated as "transfer cell" in the comparison. Participating laboratories will compare the local realization of the triple point of water and the transfer cell.

## 2. Participating Laboratories

There are nine participants in this intercomparison. Contact details are listed below in alphabetical order:

Figure 1: Pictorial view of the comparison topology





#### Table 1. Participating Laboratories

Loop 1 :

Country (code)	Laboratory	Name of contact	Address	Phone, fax, e-mail
1-Turkey (TR)	TÜBITAK UME	Mr. Ali UYTUN	TÜBITAK Ulusal Metroloji Entistüsü (UME) P.O.Box 54 Gebze, Kocaeli 41470	Phone: 90 262 6795000 /3403 Fax: 90 262 6795001 e-mail: <u>ali.uytun@tubitak.gov.tr</u>
2 – Albania (AL)	DPM	Mr.Kreshnik Hakrama	General Directorate of Metrology DPM Sami Frasheri 33 Tirana	Phone:+355 672057435 Fax:+355 4 228244 Email: <u>kreshnik.hakrama@dpm.gov.al</u>
3 – Bosnia and Hercegovina (BA)	IMBIH	Miss Nedžadeta Hodžić	IMBiH, Laboratory for temperature Dolina 6 71000 Sarajevo	Phone:+387 61 462 012 Fax:+387 33 568 909 Email: <u>nedzadeta.hodzic@met.gov.ba</u>
4 – Macedonia (MK)	вом	Ms. Olgica Petrušova	Bulevar Jane Sandanski 109a MK-1000 Skopje R.	Phone :+389 2 2403 676 Fax: +389 2 2444 677 <u>olgica.petrusova@bom.gov.mk</u>
5-Montenegro (ME)	MBM	Ms. Tanja Vukićević	Bureau of Metrology, Laboratory for temperature (ME) Kralja Nikole 2 81000 Podgorica	Phone:+382 20 601 360 Fax:+382 20 634 651 <u>tanja.vukicevic@metrologija.gov.me</u>
6 -Serbia	DMDM	Slavica Simić,	Directorate of Measures and Precious Metals (DMDM) Mike Alasa 14 11 000 Beograd	Phone: ( +381)( 66) 860 4119 <u>slavicasimic@dmdm.rs</u>



			Royal Scientific
	JNMI	Eng.	Society/Jordan National Phone: +962-6-5344701, Extension
7 -Jordan	JINIVII	Mustafa	Metrology Institute, AI- 2616
		Flaifel	Jubeiha (11941), Amman- Mustafa.flaifel@rss.jo
			Jordan

#### Loop 2 :

8 – Kingdom of Saudi Arabia	SASO / NMCC	Abdulaziz Al Zahrani	Riyadh Al Muhammadiyah -in front of King Saud University PO. B 3437 Riyadh 11471 Kingdom of Saudi Arabia	Phone:+966 11 2529999 am.zahrani@saso.gov.sa
9– United Arab Emirates	EMI	Miltiadis Anagnostou		Phone:+097124035981, miltiadis.anagnostou@qcc.abudh abi.ae

#### 3. Provisional Schedule

Each participant should complete the measurement and transport the transfer cell in about eight weeks of time period. The transport must be planned for each of the participating laboratories to the subsequent laboratory. The provisional time schedule for the comparison is given in Table 2.

If a participant anticipates difficulties in keeping the deadlines, the coordinator must be contacted immediately. In such a case the other participants will be contacted as soon as possible and be informed about eventual changes. In this situation the time table of the comparison will be revised.

Table 2	<b>2.</b> Time	schedul	e for
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	Participants and Activity	Time
UME (Turkey)		15 January-10 April 2015
For transfer cell1	IMBIH (Bosnia and Herzegovina)	17 June-14 August 2015
	MBM (Montenegro)	17 August -15 October 2015
	BOM (Macedonia)	19 October-17 December 2015



	Participants and Activity	Time	
	DMDM (Serbia)	27 May -10 July 2016	
	DPM (Albania)	24 July -12 September 2016	
	JNMI (Jordan)	14 March-16 May 2017	
For transfer cell2	EMI (United Arab Emirates)	15 November 2016- 20 January 2017	
	SASO (Kingdom of Saudi Arabia)	6 February- 10 April 2017	
UME (Turkey)	30 April -30 June 2017		
Prepare report on the comp	01 -30 July 2017		
Report on the comparison	(Final)	30 September 2017	

Deadline for reporting the result is 3 weeks after the equipment has left the laboratory. It is important that the deadline is met since the results are being analyzed continuously by the reference laboratory. If there are any problems or doubt regarding the results of the participant laboratory, the laboratory will be contacted immediately.

## 4. Description of Transfer cell and Transportation

Transfer cell was manufactured by UME in 2014. Geometrical dimensions of transfer cell are given in Table 3.

TUBİTAK UME will send transfer cell to participant 1 in a special box with ATA carnet. The box's size is 34cm x 53 cm x 33 cm and weight is about 10 kg. Each participant is responsible for sending the triple point of water cell to the following laboratory in the participant list in its special box with ATA carnet. It is crucially important that participants should make the ATA Carnet get approved at the Customs.

	Cell	Glass	h1/mm	h2/mm	D1/mm	D2/mm
Transfer cell 1	TUBİTAK UME Zero02	Borosilicate	230	315	15	50
Transfer cell 2	TUBİTAK UME Zero03	Borosilicate	232	315	15	50

Table 3. Geometrical dimensions of transfer cell	Table 3.	Geometrical	dimensions	of transfer	cell
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Where,

h1: height of the water level from the bottom of the thermometer well.

- h2: height of the cell body.
- d1: diameter of thermometer well

d2: diameter of the cell.

Upon the receipt of the transfer cell, the coordinator should be informed by e-mail. The special box should be unpacked carefully, and a visual inspection should be carried out. If the cell has any visible damage due to transportation, this must be reported to the coordinator before the comparison begins.

In the case of failure of the transfer cell, a second TPW cell is prepared and immediately provided to the participants by the pilot laboratory.

Each participating laboratory is responsible for its own costs for the measurements and transport of the TPW cell to and back from the next laboratory.

## 5. Description of Equipment used for the measurements

All participants will use two standard platinum resistance thermometers, resistance bridge, standard resistor, triple point of water cell (national reference cell), triple point of water maintenance bath and standard resistor maintenance bath for the comparison.

All participants should fill in appendix A (Description of Equipment used for the measurements).

## 6. Procedures

#### 6.1. Preparation of the triple point of water on transfer and national cell

All participants should prepare ice mantle of triple point of water cell with dry ice method.

- ✓ The thermometer well inside the transfer cell and participant national reference cell should be cleaned by inserting long metal or glass tube, which has soft cotton around their tips until no water residual left inside the cell.
- ✓ The transfer cell and participant national reference cell should be pre-cooled during several hours, typically overnight, in the water triple-point maintenance bath.
- ✓ The thermometer well should be filled to a height of about 0.5 cm with alcohol.
- ✓ The thermometer well should be filled with crushed dry ice up to the water level inside the cell using a special spoon.
- ✓ After 15-20 minutes, a 8 mm to 10 mm thickness of ice mantle is formed around the thermometer well.
- ✓ After this stage, the cell should be put into the water triple point maintenance bath.
- ✓ The thermometer well inside the transfer cell and participant's national reference cell should be cleaned with pre-cooled distilled water three times.



- ✓ The cell should be filled with pre-cooled distilled water and kept inside the water triple point maintenance bath ten days prior to measurements.
- ✓ Metal or glass rods at room temperature should be inserted into the thermometer well of the water triple point cell for a few seconds minutes to create the inner melt. When the ice mantle of the cell rotates freely around the thermometer well, then it can be assumed that the solid-liquid interface is created inside the cell and the TPW cell is ready to perform measurements.

# 6.2. Measurements of immersion temperature effect of water on transfer and national reference cell

For transfer cell and national reference cell, an immersion profile should be provided, to ensure that the measurement really senses the temperature of the ice/water interface. The measurements should be taken up to 6 cm and the step width should be 1 cm. The position of the sensor at which the comparison with the reference cell(s) was made should be indicated.

For each position, the self-heating correction should be determined and applied. Measurement should be performed at 1 mA and  $\sqrt{2}$  mA for extrapolation for 0 mA value.

All participants should fill in appendix C (Measurement results).

#### 6.3. Measurements

The temperature realized in the transfer TPW cell should be compared with the one obtained with the national reference cell of participant laboratory using standard platinum resistance thermometers (SPRTs) according to this protocol.

Transfer and reference triple point of water cells should be placed in the maintenance bath for a period of 10 day prior to the measurements. The transfer TPW cell will be compared with the reference TPW cell, at least for a total of 10 days using two different SPRTs (one cycle). Each participant must perform at least 2 cycles of measurement including a new realization of the ice mantle.

Measurements will be performed at 1 mA and  $\sqrt{2}$  mA for extrapolation for 0 mA value as mentioned before.

The triple point of water (TPW) temperature values given in this comparison result should be the values obtained after applying hydrostatic head and self-heating corrections for the measured values.

The comparison will be performed by measuring the difference in temperature between the transfer and national reference TPW cells. The difference in the observed resistances (corrected for the hydrostatic head effect and self-heating, and possible calibration of the measurement instrument) for the two cells was converted to a temperature difference using the dT/dR for the SPRT's.



#### T (national cell) - T (transfer cell) =(R (national cell) - R (transfer cell)).dT/dR

(1)

All participants will fill in appendix B (Measurement results).

#### 7. Uncertainties

The comparison is based on the measurement of the difference in temperature between the transfer cell and the national reference cell, therefore the laboratories have to evaluate the uncertainty components related exclusively to this difference.

We consider the contribution of some uncertainties to be negligible in this comparison because these uncertainties are expected to be strongly positively correlated, for the electrical measurement on the transfer and the national reference cells are made with the same bridge and practically the same ratio and also the same standard resistor stabilized in temperature is used.

So, sources uncertainties of bridge accuracy and resistor calibration and are neglected.

The uncertainty contribution from the stability of the circulating cell,  $u_{stab}$ , will be added to the value of  $u_{(Tlocal - Tcirculating)Lab}$  at the end of the circulation.  $U_{stability}$  will be evaluated through the difference between the deviation values obtained for the circulating cell at the end of the initial and final comparison with the national reference TPW temperature.

So, finally

 $U^{2}$  (T (national cell) - T (transfer cell)lab =  $U^{2}$  (T (national cell) - T (transfer cell)lab +  $U^{2}$  stability (2)

The uncertainty budget components of this comparison are given in Table 4 and all participants will fill in appendix D (Triple Point of Water Comparison Uncertainty Budget).



# Table 4. The uncertainty budget components of the comparison

Quantity	Components		
1	Chemical impurities	For national reference cell	
2	Isotopic variation	For national reference cell	
3	Repeatability for a single ice mantle (incl. bridge noise)	The repeatability for a single ice mantle is understood the experimental standard deviation of the daily obtain temperature differences between the transfer cell the national reference, divided by the square root of number of daily results.	
		✓ Same measurement procedure	
		✓ Same observer	
		✓ Same thermometer	
		✓ Same bridge	
		✓ Same maintenance bath	
		✓ Same ice mantle	
4	Reproducibility for different ice mantles and different SPRTs	The reproducibility for different ice mantles represents the additional variability introduced by measuring on several different ice mantles on transfer cell.	
		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.	
		✓ Different ice mantle	
		✓ Different SPRT	
5	Hydrostatic head of transfer cell	This is the uncertainty of the correction related to the hydrostatic pressure of transfer cell.	
6	Hydrostatic head of national reference cell	This is the uncertainty of the correction related to the hydrostatic pressure of national reference cell	
7	Perturbing heat exchanges	This component could be estimated	
		<ul> <li>✓ by comparing the deviations from expected hydrostatic pressure correction obtained in transfer and national cells (by changing immersion depth over the length of the sensor ≈ 6 cm)</li> </ul>	



	SPRT self-heating in the transfer cell and reference cell	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.
8	Others ( if any)	

#### 8. Report

The results of the comparison will be evaluated each cell from a participant will be seen as a national reference cell with a temperature assigned by its laboratory and the transfer cell compared with it. This allows calculating the temperature differences of the transfer cell from each national reference cell, and the differences thus obtained are a measure for the differences between calibrations provided by the laboratories.

Upon the receipt of the results of the measurements from all participants, the Draft A report will be prepared and circulated. If there are corrections to the draft A report to the participants are given 2 week for sending the corrections. The final report will be prepared when all the participants give approval to draft A report.

The report shall include the flowing information:

- ✓ Description of Equipment used for the measurements
- ✓ All raw data and Measurement results
- ✓ The immersion profile of transfer and national reference cell
- ✓ The uncertainty budget

Comparison will be presented the results using different possible choices of reference values which will include at least the simple and weighted mean and the median.

The publication of the results of this comparison will be shared with the participants. TUBİTAK UME plans to list the participants as co-authors, if there is general consensus on this.

The reference value of this comparison (EURAMET 1357) will be the value obtained by the pilot laboratory which participated in CCT-K7 and EURAMET.TK-7 comparisons.

At TÜBİTAK UME the national reference cell for the TPW temperature was defined by UME 92 cell which participated in CCT K7 and EURAMET.TK-7 comparisons. After these comparisons, a batch of TPW cells were compared with this national reference cell and new national reference for the



TPW temperature is defined by the average of a batch of cells. For this comparison the deviation of circulating cell from the national reference TPW temperature will be obtained and the linkage of participants of this comparison to the CCT-K7 KCRV will be established through the deviations from the circulating cell and then through the difference between the circulating cell and the UME National Reference TPW temperature.

Calculations and details will be given in the final report.

#### 9. References

- 6. **Supplementary Information for the International Temperature Scale of 1990,** *Bureau International des Poids et Mesures (BIPM) (BIPM, Sèvres Cedex, France, 1990)*
- 7. Comparison of Realizations of the Triple-Point of Water (Euromet Project No 549), Renaot E., Bonnier G., Uytun A., Ugur S., J, Tempmeko 2004, Dubrovnik, Croatia
- EURAMET.T-K7 Key Comparison of Water Triple-Point Cells, A. Peruzzi, R. Bosma, O. Kerkhof, P. Rosenkranz, M. D. Del Campo Maldonado, R. Strnad, J. Nielsen, M. Anagnostou, T. Veliki, D. Zvizdic, E. Grudnewicz, M. Nedea, D. M. Neagu, P. Steur, E. Filipe, I. Lobo, I. Antonsen, E. Renaot, M. Heinonen, T. Weckstrom, J. Bojkovski, E. Turzo-Andras, S. Nemeth, M. White, E. Tegeler, M. Dobre, S. Duris, A. Kartal Dogan, A. Uytun, V. Augevicius, A. Pauzha, A. Pokhodun, S. Simic, Int J Thermophys (2011) 32:2516–2532, DOI 10.1007/s10765-011-1048-1
- 9. Evaluation of measurement data Guide to the expression of uncertainty in measurement, *JCGM* 100:2008, *GUM* 1995 with minor corrections.



# Annex A Description of Equipment used for the measurements

Description of national reference cell:	
Manufacturer	
Type and Model	
Serial Number	
Description of Standard Platinum Resistance Thermometer (SPRT) 1	
Manufacturer	
Model	
Serial Number	
Distance from sensor midpoint to surface level of water in transfer cell	
Description of Standard Platinum Resistance Thermometer (SPRT) 2	
Manufacturer	
Model	
Serial Number	
Distance from sensor midpoint to surface level of water in transfer cell	
Description of resistance bridge :	
Manufacturer	
Model	
Serial Number	
Type of resistance bridge (AC or DC):	



# Annex A (Continuing) Description of Equipment used for the measurements

Description of standard resistor :	
Manufacturer	
Model	
Serial Number	
Description of Water triple point maintenance bath :	
Manufacturer	
Model	
Serial Number	
Stability value	
Description of Standard Resistor maintenance bath :	
Manufacturer	
Model	
Serial Number	
Stability value	
Measurement current:	
Number and sampling frequency of repeated measurements:	
Type of thermometer, length of sensor:	



#### Annex B Measurement results on first ice mantle

Date of preparation of ice mantle of transfer cell: Date of preparation of the mantle of the reference cell:

Meas.No.	Date of measurement	SPRT 1			SPRT 2			
		Difference (T(transfer)- T(nat. ref.)	Experimental standard deviation of the mean of T(transfer)	Experimental standard deviation of the mean of T(nat. ref.)	Difference (T(transfer)- T(nat. ref.)	Experimental standard deviation of the mean of T(transfer)	Experimental standard deviation of the mean of T(nat. ref.)	
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								



## Annex B (Continuing) Measurement results on second ice mantle

Date of preparation of ice mantle of transfer cell: Date of preparation of the mantle of the reference cell:

Meas.No.	Date of measurement	SPRT 1			SPRT 2			
		Difference (T(transfer)- T(nat. ref.)	Experimental standard deviation of the mean of T(transfer)	Experimental standard deviation of the mean of T(nat. ref.)	Difference (T(transfer)- T(nat. ref.)	Experimental standard deviation of the mean of T(transfer)	Experimental standard deviation of the mean of T(nat. ref.)	
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								



#### **Annex C: Immersion profile**

Immersion profile (for transfer cell)

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

Immersion profile (for national reference 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.



# Annex D Triple Point of Water Comparison Uncertainty Budget

Quantity	Components	Standard	Sensibility	Uncertainty
		uncertainty	coefficient	contribution
Qi		U(Qi)		u <sub>i</sub> in mK
1	Chemical impurities			
2	Isotopic variation			
3	Repeatability for a single ice mantel (incl. bridge noise)			
4	Reproducibility for different ice mantles and different SPRT			
5	Hydrostatic head of transfer cell			
6	Hydrostatic head of national reference cell			
7	Perturbing heat exchanges			
8	SPRT self-heating in the transfer cell and reference cell			
9	others			
Combined uncertainty				
Expanded uncertai	inty			