XVII IMEKO World Congress
Metrology in the 3rd Millennium
June 22–27, 2003, Dubrovnik, Croatia

CONSTRUCTION AND CHARACTERISATION OF THE UME MADE WATER TRIPLE POINT CELLS

Ali Uytun, Aliye Kartal Dogan and Sevilay Ugur

TUBITAK –National Metrology Institute (UME), Kocaeli, Turkey

Abstract – The International Temperature Scale of 1990 (ITS-90) specifies triple, melting and freezing points to realize the scale [1]. One of the major temperature fixed points in the ITS-90 scale is the triple point of water (TPW), at which temperature solid, liquid and vapour phases of pure water coexist in thermal equilibrium. This temperature has been assigned a value of 273,16 K (0,01°C) on the ITS-90. It is the fundamental definition of the fixed point of the ITS-90 scale and the one defining fixed point of the Kelvin thermodynamic temperature scale.

UME Temperature Laboratory has been constructing TPW cells since 1992. This paper describes the construction processes (annealing, cleaning and filling) of the TPW cell, its realization and finally the results of EUROMET comparison of TPW cells.

Keywords: TPW, realization, comparison

1. INTRODUCTION

The temperature unit, Kelvin, is defined as follows using triple point of pure water. "The Kelvin, unit of thermodynamic temperature, is the fraction 1/273,16 of the thermodynamic temperature of triple point of water (TPW). The temperature at which solid, liquid and vapour phases of pure water coexist in thermal equilibrium. This temperature has been assigned a value of 273,16 K (0,01°C).

The triple point of water is required for all realizations of the ITS-90 in ranges and sub-ranges for which standard platinum resistance thermometers (SPRT's) are specified. The triple points of water cells are used for calibrating SPRT's. They are used to determine the resistance ratio (W_{t90}) defined in the ITS-90 as resistance at temperature t_{90} divided by resistance value at 0,01°C. Measurements of R at TPW (0,01°C) provide also check and record of the stability of SPRT's.

2. CONSTRUCTION

UME constructs triple point water cells in cylindrical shape borosilicate glass, with a bore re-entrant tube, which serves as a thermometer well.

Construction process involves three steps namely, cleaning, annealing and filling.

The cleaning of borosilicate tubes consist of washing in soapy-hot water and rinsing with water several times, then soaking in a acid mixture for a day and then rinsing with distilled water several times.

The cleaned and labeled borosilicate TPW cell is annealed at high temperatures to remove the contaminations on the surface of the cell.

Cells are filled with the 3-distilled water. Just before filling, steam is passed through the cell for final cleaning. When the amount of water inside the cell reaches approximately 500-750cm^3, the cell is sealed.

The UME made triple points of water cells are produced in three different sizes. The geometrical dimensions and specifications are shown in Table I.

Table I. The specifications of UME made TPW cells

Specifications	Thin -Short	Thin - Long	Wide
Dimensions	42mm O.D. X 360 mm long, 11 mm I.D.	42mm O.D. X 410 mm long, 11 mm I.D.	60mm O.D. X 390 mm long, 11 mm I.D.
Immersion Depth *	230 mm	280 mm	270 mm
Reproducibility	0,00001°C	0,00001°C	0,00001°C
Uncertainty	< 0,0001°C	< 0,0001°C	< 0,0001°C

* Height of the water level from the bottom of the thermometer well.

3. PREPARATION OF CELLS FOR MEASUREMENTS

The dry ice method and immersion cooler method have been used regularly for the realisation of the cells at the UME Temperature Laboratory [2].

3.1. Dry ice method

The thermometer well inside the triple point of water cell is cleaned by inserting long metal or glass tube, which has soft cotton around their tips until no water residual left inside the cell. Prior to beginning of an ice mantle generation around the thermometer well, the cell is pre-cooled by inserting the cell inside a water triple point maintenance bath for two hours. The thermometer well is filled with crushed dry ice up to the water level inside the cell. After 15-20 minutes, a 5 mm to 8mm thickness of ice mantle is formed around the thermometer well. After this, the cell is returned into the water triple point maintenance bath and, kept at temperature of 0.006 (± 0.001) ^{o}C.

3.2. An immersion cooler method

An initial state of preparation of cell is the same as explained above procedure; only difference is that, the way of preparation of an ice mantle formation around the thermometer well. An immersion cooler apparatus is used to prepare an ice mantle inside the cell. This is 420mm long, 8mm diameter and filled with refrigerator liquid [3]. After the initial stage of preparation of the cell, the thermometer well of the cell is filled with cooled ethanol until the water level inside the cell. Before inserting the immersion cooler, one or two drops of alcohol are dropped into the thermometer well. Then, a few number of crushed dry ice is added into the cell to initiate the ice mantle at the bottom of the cell. After sufficient thickness of ice mantle generated, cooled alcohol is poured into the thermometer well. An immersion cooler is then inserted into the cell. After 40-50 minutes, 5mm-8mm thickness of an ice mantle is formed. The immersion cooler is then taken out from the cell. Ethanol inside the cell is pouring out and the thermometer well is rinsed out several times with cooled distilled water.

After preparing the ice mantle of the cell, it is returned into the water triple point maintenance bath, which is kept at temperature of (0,006 ± 0,001) ^{o}C. The cell is filled with cooled distilled water and kept inside the water triple point maintenance bath at least two full days prior to measurements. Metal or glass rods at room temperature are inserted into the thermometer well of the water triple point cell for a few seconds to create the inner melt. When the ice mantle of the cell rotates freely around the thermometer well, it is understood that the solid-liquid interface is created and the TPW cell is ready to make measurements.

The UME built and prepared triple point of water cell is shown in figure 1.

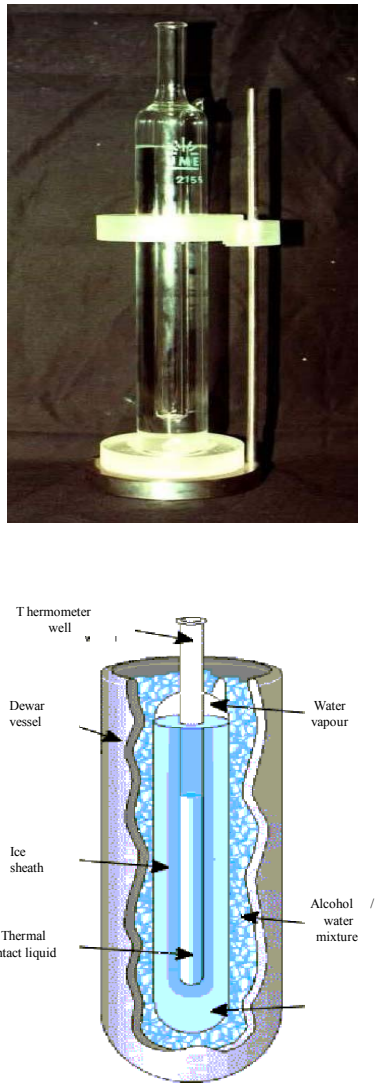

Fig. 1. The UME built and prepared triple point of water cell

4. MEASUREMENTS

The newly made TPW cells are always compared with the UME reference TPW cell. The F18 AC and MI6015T DC resistance bridges are used for all comparison measurements. Different manufacturer types of SPRT's are used in an attempt to eliminate the mechanical shock effects on the thermometers during comparison measurements of the cells. Reference resistors (Tinsley 25Ω and 100Ω) are also maintained at a temperature (20,00±0,02) ^{0}C.

The measurement set up of the TPW comparison is shown in figure 2.

Comparison measurements are performed at two different current (1 mA and √2 mA) values to extrapolate for 0 mA value according to the following equation:

$$R_{0mA}(t90)=2R_{1mA}(t_{90}) - R_{\sqrt{2}\,mA}(t90) \qquad (1)$$

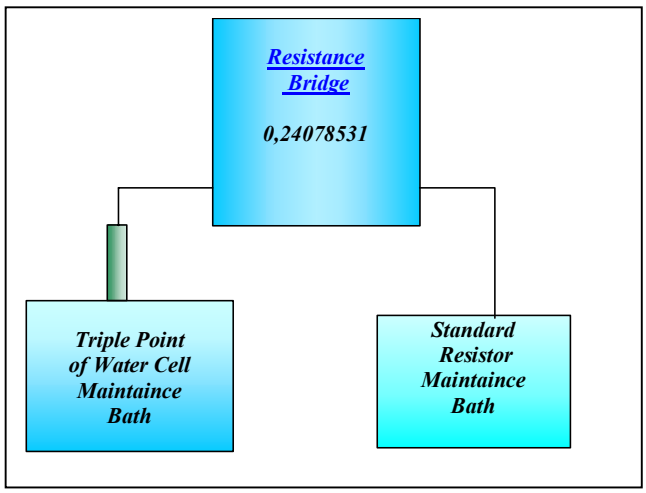

Fig.2. The measurement set up the TPW cell comparison

The equilibrium temperature of a cell is repeatable within ±0,0001 °C of the mean equilibrium temperature. The typical triple point plateau of TPW cell is shown in Figure 3. The standard deviation and duration of the plateau is approximately 0,05 mK and 12 hours respectively.

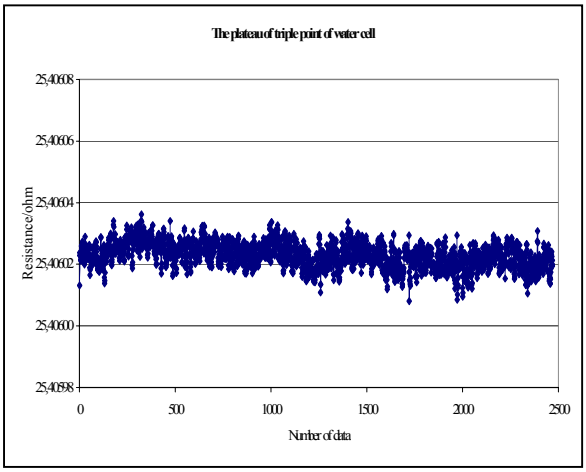

Fig. 3. The plateau of triple point of water cell

Figure 3 shows the results of UME measurements on the typical UME cell. The total time in the figure is more than 12 hours and the small drift, which is apparent in the figure, is due to disappearing of the liquid phase around the re-entrant tube and conversion to the solid phase.

The immersion test is done to new made TPW cell by taking out the SPRT from the bottom of the cell. The immersion test of cell is shown in figure 4. The temperature difference up to 6 cm from the bottom is approximately 0.1mK.

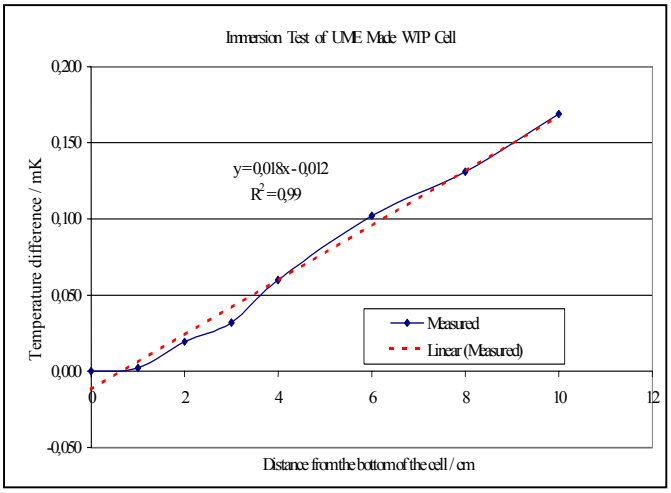

Fig. 4. The immersion test of triple point of water cell

UME Temperature laboratory has participated EUROMET comparisons (Project 278 and 549) in recent years. The results of these comparisons are shown in Table II [4,5].

Table II. The results of comparisons

Cells	Comparisons	Year	Difference mK
UME 4	EUROMET 278	1997	0,090
UME 6	EUROMET 278	1997	0,081
UME 13	EUROMET 278	1997	0,080
UME 52	CCT2000-14	2000	0,040

5. CONCLUSIONS

UME has been constructing TPW cells for more than 10 years. Within this time interval these cells have been used either for comparisons or realizing the ITS-90 Scale at UME. All these measurements have shown that the uncertainty <0,1 mK is easily achievable.

REFERENCES

[1] H. Preston-Thomas, "The International Temperature Scale of 1990 (ITS-90)", *Metrologia*, vol. 27, pp. 3-10, 1990.

[2] A.T. Ince, A. Kartal, "Realization of Home Made Water Triple Point Measurements Using Immersion Cooler and Dry Ice Methods and Their Respective Differences", *XIV IMEKO World Congress*, vol. 6, pp. 203-205, 1997.

[3] J.P. Evans, D.M. Sweeger, "The review of the Scientific Instruments", 40, 2, pp 376-377, 1969.

[4] E. Renaot, M. Elgourdou, G. Bonnier, "Interlaboratory comparison of water triple-point cells realizations", *Metrologia*, vol. 37, pp. 693-699, 2000.

[5] R. Pello, R. Köhler, "Comparison of Water Triple-point Cells from different Manufacturers, CCT/2000-14, 2000.

Authors:
Ali Uytun
TUBITAK –National Metrology Institute (UME),
Kocaeli, Turkey,
Phone:+90(262)646 63 55
Fax: +90(262)646 59 14
ali.uytun@ume.tubitak.gov.tr

Aliye Kartal Dogan
TUBITAK –National Metrology Institute (UME),
Kocaeli, Turkey,
Phone:+90(262)646 63 55
Fax: +90(262)646 59 14
aliye.kartal@ume.tubitak.gov.tr

Dr. Sevilay Ugur
TUBITAK –National Metrology Institute (UME),
Kocaeli, Turkey,
Phone:+90(262)646 63 55
Fax: +90(262)646 59 14
sevilay.ugur@ume.tubitak.gov.tr