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WATER TRIPLE POINT CELLS COMPARISON

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ABSTRACT

This work, which consists of a comparison between three water triple point (WTP) cells available at INMETRO, was developed to compare our measurements with those obtained by the Bureau International des Poids and Measures (BIPM) and to check whether the cells are in harmony with its values. This work also describes the equipments employed during these measurements and relates the procedures and methodology followed to compare the cells. Tables and graphics from data are also shown.

1. INTRODUCTION

The triple point of water (0,01°C) is the most important fixed point in the International Temperature Scale 1990. The temperature in this scale is measured in terms of ratio R(T)/R(0,01°C) with a resistance thermometer; consequently, it is important to know the accuracy of the measurements at the WTP and the reproducibility of the WTP for, above 0,01°C, any errors in R(0,01°C) are amplified in the resistance ratio R(T)/R(0,01°C).

In the beginning of 1996 the Water Triple Point Cell 494 was sent to the BIPM in order to participate in an international comparison between WTP cells. With this work we were able to check the reproducibility of WTP cells from INMETRO as well as the harmony of the measurements performed in INMETRO in relation to BIPM.

The following data refer to comparison measurements taken between the water triple point cells 494, 483 and 221 on August, 30th before the cell 221 was also sent to the BIPM.

2. MEASUREMENT EQUIPMENT

To perform the measurements it was used an AC Bridge from AUTOMATIC SYSTEM LABORATORY model F18 with a standard resistor from H. Tinsley (100 ohms, s/n 236063) immersed in a controlled oil bath from Guildline model 9732VT. During the measurements the temperature in the oil bath was 20,01 °C ± 0,01°C.

The comparison between the cells was performed by a 25 ohms standard platinum resistance thermometer from Tinsley model 5187A s/n 238698 (SPRT).

In order to check the stability of the SPRT, its resistance was measured at the gallium melting point before and after the WTP measurements.

3. PROVENANCE OF THE CELLS

The WTP cell ISOTECH s/n 494 was compared to other two triple point cells available at INMETRO. One of them, cell 221, was given by the Physikalisch-Technische Bundesanstalt and the other (ISOTECH s/n 483) was purchased together with ISOTECH s/n 494 from its representatives in Brazil. Both cells from ISOTECH were certified with an uncertainty of 0,1 mK with a confidence level of 95% by NPL on December 14th, 1990.The cell from PTB was certified with an uncertainty of 0,2 mK in November 1978 The certificates provided by NPL and PTB do not mention the water isotopic composition

The gallium cell was manufactured by Engelhard Pyro Controle and certified by Laboratoire National d'Essais in July 1991. The declared value in this certificate is 29,7652 ± 0,0017 °C with a confidence level of 3s.

4. WTP STORAGE APPARATUS

The cells were immersed in water triple point bath from ISOTECH. This bath contains about 34 litters of water which is stirred with air supplied by a pump. The temperature of the water is maintained at 0,010°C by four thermoelectric cooling modules. The current through the cooling modules is controlled by a proportional controller which uses a precision, high-stability thermistor probe to sense bath temperature.

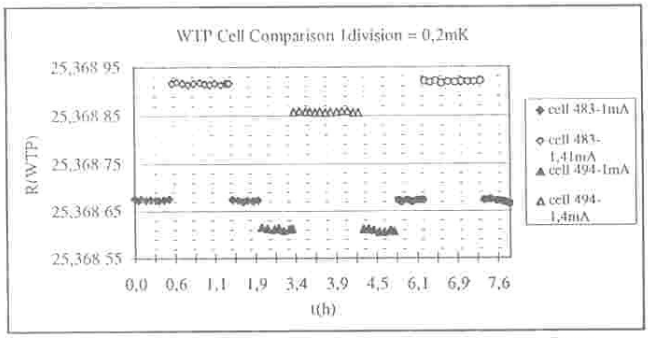
5. PROCEDURE TO BUILD THE MANTLE.

The mantle was prepared inserting repeatedly a liquid nitrogen cooled rod into the well of the triple point cells which contained some alcohol. Once the mantle was ready and the well of the cell cleaned, a small amount of new pre-cooled water was poured into it to improve the thermal contact between the cell and the thermometer. The WTP cell was allowed to rest for 24 hours before an acrylic rod was introduced into the well in order to melt a thin layer of ice next to it.

6. METHODOLOGY AND RESULTS

The gallium melting point was measured before and after the water triple point measurements, in order to check the thermometer stability. A 25-ohm platinum resistance thermometer from Tinsley was employed to perform the measurements of the three cells. The thermometer was left for 30 minutes in every cell before the measurements started. The cell sent to BIPM (ISOTECH s/n 494) was compared to two other WTP cells available at INMETRO: ISOTECH s/n 483 and PTB s/n 221. The sequence of measurements was the following: firstly the cell from ISOTECH 483, secondly ISOTECH 494 and thirdly PTB 221.

The values measured by the bridge ASL F18 were taken automatically at regular intervals for a period of about 30 minutes for 1 mA and 1,4 mA currents. These values were sent to an interfaced computer and stored in a file. The program developed at INMETRO only takes into account the last of three successive measurements in equilibrium taken by the bridge. This way we had at least 131 good values measured for every current. These data can be seen in graphic 1



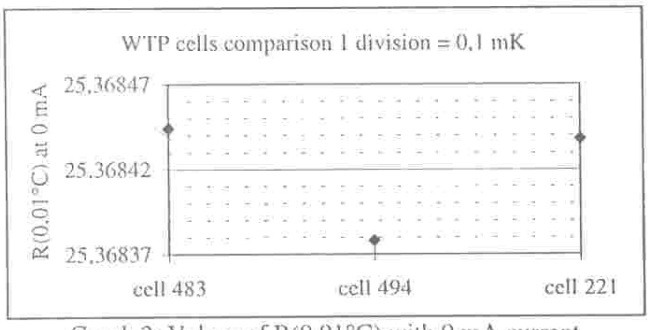
Graphic 1: Measured values of WTP comparison

The measurement data plotted on graph 1 were treated in order to get the average values for each cell of the resistance at the definition point of the triple point of water (0,01°C) applying hydrostatic head and self heating corrections. All measurements were taken on the same day. The values are shown in table 1 below.

Table 1: Measurement results of WTP cells comparison in resistance

	R(0,01°C) at 0 mA		
date	cell 483	cell 494	cell 221
30/08/96	25,368444	25,368378	25,368438

In the following graphic the values shown in table 1 are plotted:



Graph 2: Values of R(0,01°C) with 0 mA current

In table 2 below are shown the corresponding differences in milikelvin between the WTP cells from table 1.

Table 2: Measurement results of WTP cells comparison in mK

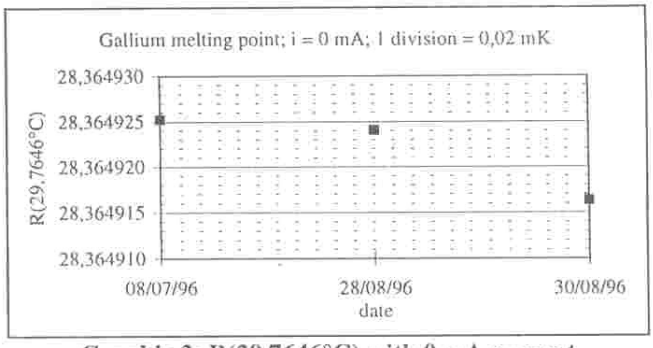
	cell 221 - cell 483	cell 483 - cell 494	cell 221 - cell 494
Difference in mK	-0,053	0,644	0,591

Each value shown in table 3 below corresponds to an average resistance calculated at the definition point of gallium melting point (29,7646°C) after hydrostatic effect and self heating corrections. The measurements were taken on July 8th, August 29th and August 30th.

Table 3: Measurement results of stability of the SPRT in resistance

	R(29,7646°C) at 0 mA		
date	08/07/96	29/08/96	30/08/96
R (Ω)	28,364 925	28,364 924	28,364 916

In the following graphic are plotted the values shown in table 3:



Graphic 3: R(29,7646°C) with 0 mA current

In table 4 below are shown the corresponding differences in milikelvin at gallium melting point performed on different days. The gallium melting point on August 29th was performed just before the measurements in the WTP had begun. The gallium melting point on August 30th was performed just after the measurements in WTP.

Table 4: Measure of SPRT stability in mK

	R(29,7646°C) at 0 mA	
date	from: 30/08/96 to: 08/07/96	from: 30/08/96 to: 29/08/96
Δ in mK	-0,089	-0,077

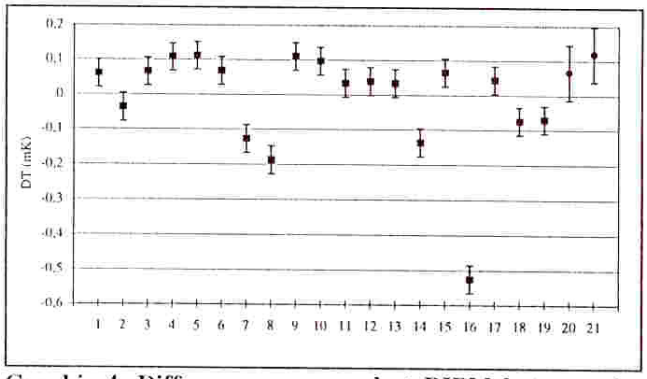
In the following table are shown the results of the international comparison between the water triple point cells [2]. To this table we added the results of these measurements, i.e., the difference corresponding to cells 221 and 483 in relation to the BIPM cell. These two last values were calculated according the following equation

$$T_{cell} - T_{ref} = \left(T_{cell} - T_{494}\right) + \left(T_{494} - T_{ref}\right)$$

Table 5: Temperature differences between the WTP cells of groups 1,2 and 3 and the BIPM WTP reference cell.

Nr	Cell	Tcell - Tref (mK)
1	NPL 723	0,060
2	NPL 712	-0,037
3	ETL J2	0,065
4	1984(NIST-type)	0,107
5	2011(NIST-type)	0,111
6	IMGC 34	0,068
7	VNIIM 2R	-0,128
8	VNIIM 4R	-0,188
9	KRISS 1	0,109
10	KRISS 2	0,096
11	MSL 82/2	0,033
12	MSL 84 7	0,038
13	IPQ 033	0,033
14	IPQ 299	-0,137
15	SISIR	0,064
16	INMETRO 494	-0,526
17	NMI 94T214	0,043
18	NMI 89T084	-0,075
19	INM Rom.	-0,071
20	INMETRO 221	0,065
21	INMETRO 483	0,118

These data are plotted on the following graph



Graphic 4: Differences measured at BIPM between the WTP cells of groups 1, 2 and 3 and the BIPM WTP reference cell (BIPM uncertainty ± 0,04°C, k = 1) and differences measured at INMETRO (uncertainty ± 0,08°C, k = 1) between 221 and 483 and the BIPM WTP reference cell.

7. UNCERTAINTIES OF THE RESULTS

The comparison consisted of measuring the difference in temperature between three different WTP cells with the same SPRT and bridge. Therefore we took into account the following uncertainties components:

Type A:

⇒ S_R - uncertainty due to the repeatability of measurement results under the following conditions:

⇒ Same bridge

⇒ Same platinum resistance thermometer

⇒ Same isothermal enclosure to the WTP cells

⇒ One ice mantel realization (measurement over a short period of time)

Type B:

As the measurements were taken in a short period of time we did not take into account the uncertainty of the standard resistance in the evaluation of type B uncertainties. We considered the possible long term drift have negligible influence.

⇒ U_B - uncertainty due to electrical measurement as stated by bridge manufacturer

⇒ U_{HHC} - uncertainty due to hydrostatic head

⇒ U_{SHC} - uncertainty due to self heating correction

⇒ U_{SPRT} - uncertainty due to stability of the SPRT measured at gallium point. This was considered as the change in R(29,7646°C) observed from 29th to 30th of August, 1996.

In the table below are shown the contributions of each uncertainty.

Table 6: Uncertainty budget

symbol	**value ±**	**Ci**	**Ui (mK)**	**f.d.**
S_R	2 E-7 Ω	10000 mK/Ω	0,002	131
U_B	0,2/√3 ppm	0,2537 mK/Ω	0,029	∞
U_{HHC}	28 E-7 K	1000 mK/K	0,003	∞
U_{SHC}	0,064 mK	1	0,038	∞
U_{SPRT}	0,044 mK	1	0,044	∞
Combined uncertainty		(k=1)	0,083	
Expanded uncertainty		**(k=2)**	**0,163**	

8. CONCLUSIONS

Table 2 shows the differences between the cells ISOTECH 494 and the other two available at INMETRO (-0,6 mK) are about the same as those found by BIPM as compared to BIPM WTP cell (-0,53 mK).

Table 4 shows a diference of 7,7 μΩ (equivalent to 0,08 mK) at gallium melting point from 29th to 30th of August. This diference might be due to small drifts in the thermometer resistance or imperfections in the gallium point realization.

Table 5 shows the differences between the other two WTP cells available at INMETRO are about the same as those found by the BIPM for other countries as compared to BIPM WTP cell.

Graphic 4 shows that the WTP cell ISOTECH 494 is defective. This failure might have been caused by the isotopic composition of the water. This graphic also shows

that the differences found by the IMETRO are about the same as those found by the BIPM. Therefore, we can perform calibrations of SPRT with a good confidence level using the other cells.

The difference between the cell 221 and the cell from BIPM calculated by the INMETRO was found to be 0,065 mK with an uncertainty of 0,08 mK (table 5). This result is compatible with the difference found at BIPM on november 28th which is 0,051 mK with uncertainty of 0,04mK.

9. REFERENCES

1 - Supplementary Information for the International Temperature Scale of 1990, 1990, BIPM.

2 - Report on the International Comparison of Water Triple Point Cells, R. Pello, R. Goebel and R. Köhler, 1996, BIPM.