ITS-90 REALIZATION IN THE RANGE FROM –190°C TO 420°C AT INMETRO

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ABSTRACT

Since the 90's, Inmetro (Brazil) has performed bilateral comparisons of ITS-90 realisations in the range from –190°C to 420°C aiming to evaluate its fixed-points cells and procedures. In the past comparisons realised by Inmetro, sealed cells were used.
In a bilateral comparison with PTB (Germany) performed during 2001and 2002, the differences found were most significant at the Ar, Zn and Sn fixed points.
In 2002, Inmetro qualified its Sn and Zn open cells, which temperatures appeared to be closer to the ITS-90 values than similar sealed cells. As a result, Inmetro performed another bilateral comparison with PTB, when these indications could be confirmed.
In addition, it was investigated the reason for the differences presented at the Ar triple point, because both Institutes (Inmetro and PTB) have exactly the same apparatus, despite of the results had been corrected for any systematic influence due to the Inmetro realisation procedure.
The main aim of this work is to confirm the Inmetro best measurement capability in the range from –190 °C to 420°C considering all bilateral comparisons performed.

1. INTRODUCTION

The calibration of long stem standard platinum resistance thermometers (SPRTs) by fixed point method is an important activity on the realization and dissemination of the International Temperature Scale of 1990 (ITS-90) [1,2].

One of the main components of SPRT calibration uncertainty budget is due to the impurities of the fixed cell reference material. To evaluate it, preliminary it is advisable to know the purity of material. A second step is the comparison of cells.

Although Inmetro had recently developed its own cells for the triple points of water and mercury, most calibrations were performed using commercially available fixed-point cells. Therefore, following the goal of to better determine its uncertainties, Inmetro has been performing comparisons of the ITS-90 realizations with different NMIs. Sometimes it was also performed direct comparison of cells, which consists on realization of both fixed points simultaneously [3].

In a comparison using fixed point cells, temperature measurements are taken with a SPRT at the reference cell and test cell sequentially to determine the temperature difference between them. This difference can be determined directly by the change in the W value (Rt/Rwtp) for the reference fixed point or by change in the resistance value.

On the other hand, in a comparison of the ITS-90 realizations, a SPRT is calibrated in two different cell sets. Initially the SPRT is calibrated in a reference cell set, after that it is calibrated at the test cell set. Finally, it is calibrated in the reference set a second time, in order to check if any deviations has appeared during the process and the difference of the cells are defined by changes in the W values only. This type of comparison is the realization of the ITS-90 over the temperature range in which the SPRT was calibrated and it is very important to use only stable SPRTs. The measurements were corrected for

immersion depth and self-heating, but not for cell pressure because it is not possible to access the pressure of the sealed cells. So, this work will present these two kinds of comparisons and its influence on the Inmetro uncertainty budget for the realization of the ITS-90 over the range –190°C to 420°C.

2. EQUIPMENT

Inmetro has sealed and open cells. The fixed points are realized using three zone furnaces for indium, tin and zinc points (manufactured by Hart). The gallium point is performed in a furnace supplied by Isotech or a home made bath. The mercury triple point is performed in one cryostat supplied by Isotech and the argon triple point in an apparatus supplied by SORIME, developed by INM-France.

The water triple point (WTP) cells normally are kept in a dewar flask with crushed ice. After the ice mantle realization using CO_2 (crushed dry ice), the WTP cell is inserted in PVC tubes and conserved in ice for at least two months.

All measurements with SPRTs are done automatically using an AC automatic resistance bridge ASL, model F18 controlled by a computer, which also performs the data acquisition. The standard resistors are maintained in an oil bath at 20 °C ±0.01 °C.

The sealed cells of indium, tin and zinc were supplied by Engelhard-Pyro Control and the open cells of tin and zinc were manufactured by Leeds and Northrup. The gallium and the mercury cells were supplied by Isotech and the argon triple point was made by INM-France.
The water triple point cells used in those comparisons were supplied by CENAM and Hart Scientific.

3. RESULTS OF PAST COMPARISONS

Inmetro evaluated some cell temperature differences on previous bilateral cell comparisons performed between Inmetro and CENAM-Mexico (in 1997, range: –39 °C to 420 °C) [4] and between Inmetro and NRC-Canada (in 2000, range: –190 °C to 962 °C) [5].
The results of these comparisons are shown on Table 1:

TABLE 1: Results from fixed point cell comparisons: Difference between Inmetro and CENAM in 1997 [4], Inmetro and NRC in 2000 [5]. Uncertainties are given for $k = 2$.

Fixed Point	T (Inmetro) – T (CENAM) / mK	T (Inmetro) – T (NRC) / mK
Zn (EPC 027)	–1.52 ± 0.52	–1.3 ± 1.2
Sn (EPC 032) (EPC 047)	–1.85 ± 0.21[a]	–1.9 ± 0.6[a] 0.12 [b]
In (EPC 046)	– 0.23 ± 0.18	– 0.3 ± 0.6
Ga (s/n 168)	– 0.17 ± 0.06	– 0.1 ± 0.6
Hg (M 036)	– 0.14 ± 0.18	– 0.4 ± 0.6
Ar (INM 031)		0.8 ± 1.0[c]

[a]Sn sealed cell s/n EPC 032
[b]Sn sealed cell s/n EPC 047
[c]The uncertainty is an estimate based on experience [5]

In the years 2001-2002 a bilateral comparison in the temperature range from –190 °C and 420 °C was organised between Inmetro and PTB [6]. A standard platinum resistance thermometer (SPRT) of 25 Ω

was calibrated at the temperature fixed points of Ar, Hg, the triple point of water (TPW), Ga, In, Sn and Zn, following the EUROMET K3 comparison protocol. The results are shown in table 2:

TABLE 2: Results of the ITS-90 realization comparison between Inmetro and PTB (in the years 2001-2002), using a long stem SPRT Rosemount model 162 CE

Fixed point	Zn	Sn	In	Ga	Hg	Ar
T (Inmetro)−T (PTB) /mK	−4.13 ±2.54	−2.26 ±1.43	−1.09 ±1.12	−0.32 ±0.33	−0.06 ±0.80	1.90 ±1.27

According to these results the temperature differences for zinc, tin and argon points are not covered by the expanded uncertainties. Especially for zinc and tin sealed cells, the temperatures are below the reference cells (see table 1) and the comparison performed between Inmetro and PTB in 2001-2002 showed that those differences are even higher.
As a result, Inmetro decided to qualify its zinc and tin open cells, performing another comparison with PTB, in order to verify those temperature differences.

4. ZINC AND TIN CELLS COMPARISON AT INMETRO

During the years 2001, 2002 and 2003 Inmetro compared its open and sealed cells, either by direct or indirect method [3], evaluating the existing temperature differences.
In table 3 and 4 the temperature differences between tin sealed cells (EPC 032 and EPC 047) and zinc sealed cells (EPC 027 and EPC 033) are shown.

TABLE 3: Results of tin sealed cells comparison performed at Inmetro in 2001 (directly), using a L&N long stem SPRT, model 8163B.

Date	9-jan-01	10-jan-01	11-jan-01	12-jan-01	Average
Cell EPC 032 (Sn) -- R/ Ω	48.371 461	48.371 463	48.371 474	48.371 462	48.371 465
Cell EPC 047 (Sn) -- R/ Ω	48.371 573	48.371 585	48.371 582	48.371 575	48.371 579
Cell dif. (EPC032-047)/ mK	-1.12	-1.22	-1.08	-1.13	-1.14 ±0.06

TABLE 4: Results of zinc sealed cells comparison performed at Inmetro in 2001 (directly), using a L&N long stem SPRT, model 8163B.

Date	16-jan-01	17-jan-01	18-jan-01	19-jan-01	Average
Cell EPC 033 (Zn) -- R/ Ω	65.647 120	65.647 156	65.647 170	65.647198	65.647 161
Cell EPC 027 (Zn) -- R/ Ω	65.647 179	65.647 190	65.647 222	65.647241	65.647 208
Cell dif. (EPC033-027)/ mK	-0.59	-0.34	-0.52	-0.43	-0.47 ±0.11

According to the results in table 1, the temperature difference between tin cells EPC 032 and EPC 047 is –2.02mK (an indirect comparison between those cells using the results of the NRC-Inmetro comparison).

During the years 1997 to 2001, a long stem SPRT L&N model 8163B was used as a check thermometer on the ITS-90 realizations at Inmetro. This SPRT was the same thermometer used to perform direct comparisons shown in tables 3 and 4. Using the W values obtained in tin sealed cells (EPC 032 and EPC 047) and zinc sealed cells (EPC 033 and EPC 027) it was possible to compare them indirectly. The results are shown in tables 5 and 6.

TABLE 5: Results of tin cells comparison performed at Inmetro during the years 1997 to 2001 (indirectly), using a L&N long stem SPRT, model 8163B.

	WSn EPC 032	WSn EPC 047
Average =	1.892 542 3*	1.892 547 4**
Std.dev./mK =	0.29	0.21
Cell difference (EPC 032 – EPC 047) /mK =		-1.39

* seven realizations for EPC 032 cell; ** eight realizations for EPC 047 cell.

TABLE 6: Results of zinc cells comparison performed at Inmetro during the years 1997 to 2001 (indirectly), using a L&N long stem SPRT, model 8163B.

	WZn EPC 033	WZn EPC 027
Average =	2.568 456 6*	2.568 456 6**
Std.dev./mK =	0.34	0.45
Cell difference (EPC 033 – EPC 027) /mK =		-0.01

* sixteen realizations for EPC 033 cell; **thirty one realizations for EPC 027 cell.

The results below show the temperature difference between tin sealed cell EPC 047 and tin open cell L&N s/n 742876 and the temperature difference between zinc sealed cell EPC 027 and zinc open cell L&N s/n 742879.

TABLE 7: Results of tin cells comparison performed at Inmetro in 2002-2003 (indirectly), using a Hart long stem SPRT, model 5681.

	WSn EPC 047	WSn L&N
Average =	1.892 698*	1.892 708**
Std.dev./mK =	0.17	0.16
Cell difference (EPC 047 – L&N) /mK =		-2.78

* three realizations for EPC 047 cell; ** four realizations for Sn L&N cell.

TABLE 8: Results of zinc cells comparison performed at Inmetro in 2002-2003 (indirectly), using a Hart long stem SPRT, model 5681.

	WZn EPC 027	WZn L&N
Average =	2.568 728*	2.568 736**
Std.dev./mK =	0.15	0.07
Cell difference (EPC 027 – L&N) /mK =		-2.43

* seven realizations for EPC 027 cell; ** eight realizations for Zn L&N cell.

5. ITS-90 REALIZATION COMPARISON BETWEEN INMETRO AND PTB IN 2002

In order to check the temperature differences found between its sealed and open cells, Inmetro proposed another bilateral comparison with PTB over the range –190°C to 420°C. PTB was the pilot laboratory of this comparison, performed in 2002, calibrating a SPRT Hart model 5681 after and before Inmetro in following fixed points: argon, mercury, gallium, indium, tin and zinc. The results are shown in tables 9 and 10.

TABLE 9: Results of the ITS-90 realization comparison between Inmetro and PTB (in 2002), using a long stem SPRT Hart model 5681.

Fixed point	Zn	Sn	In	Ga	Hg	Ar
Initial difference (*T INM – T PTB*)/ mK	-1.02	-0.09	-1.22	0.05	-0.30	0.71
Final difference (*T INM – T PTB*)/ mK	-0.71	0.22	-0.78	0.13	-0.34	
Mean difference (*T INM – T PTB*)/ mK	-0.86	0.07	-1.00	0.09	-0.32	0.71

TABLE 10: The mean differences of the ITS-90 realization comparison between Inmetro and PTB in 2002.

Fixed point	Zn	Sn	In	Ga	Hg	Ar
(*T INM – T PTB*)/ mK	-0.86 ±2.56	0.07 ±1.45	-1.00 ±1.39	0.09 ± 0.34	-0.32 ± 0.80	0.71 ± 1.27

6. DISCUSSION

The direct comparison of tin sealed cells performed at Inmetro in 2001 showed that the temperature of tin cell EPC 047 is higher than the temperature of tin cell EPC 032 (see table 3, where *T*.EPC 032 – *T*.EPC 047 = –1.14 mK). This tendency was confirmed by the comparison with NRC, which can be seen in table 1, and it is in agreement with the mean value of the comparisons performed during years 1997-2001, as shown in table 5. However, when the tin sealed cell EPC 047 was used in the bilateral comparison between Inmetro and PTB in the years 2001-2002, the temperature difference between Inmetro and PTB at the tin point was (–2.26 ±1.43) mK (table 2). As a consequence it was decided to compare this cell with another tin open cell from L&N.

The ITS-90 realization at Inmetro, using a tin open cell showed that the temperature difference between tin sealed cell EPC 047 and tin open cell L&N s/n 742876 is –2.78 mK. The temperature diference between the tin open cell L&N s/n 742876 and the PTB tin reference cell is (0.07 ± 1.45) mK (table 10). Linking this result with the previous result, the temperature difference between EPC 047 cell and PTB tin reference cell is –2.34 mK + 0.07 mK = –2.27 mK, confirming the temperature difference mentioned above.

Linking the results obtained in two bilateral comparisons between Inmetro and PTB to zinc point, it was found the following: $T(INM)_{EPC\ 027} - T(PTB) = (-4.13 \pm 2.54)$ mK and $T(INM)_{L\&N} - T(PTB) = (-0.86 \pm 2.56)$ mK. Then, $T(INM)_{EPC\ 027} - T(INM)_{L\&N} = -4.13$ mK + 0.86 mK = –3.27 mK. This last result is quite close to the result shown in table 8 (–2.43 mK).

Concerning the triple point of argon realization, the compatible result reached in the last comparison with PTB show that, probably the difference in the immersion depth used before was the responsible for it. After the end of the measurements, it was found that a wrong value for the thermometric well depth was considered, which led to an immersion error of the thermometer of 20 to 30 mm. This fact can explain the results obtained, which showed slightly higher temperatures for the measurements performed at Inmetro when compared to those at PTB, for Ar triple point.

7. CONCLUSIONS

The ITS-90 realization at Inmetro using open cells for zinc and tin fixed points is, in comparison with the realization at PTB, covered by the corresponding expanded uncertainties (for *k=2*). The results reached in the last bilateral comparison with PTB and performed through internal comparison between the open and sealed cells show it.

Although the results obtained with indirect cell comparisons were compatible, the standard deviation is still high, specially for zinc and tin fixed points along time. So, it indicates that the procedures of checking point and fixed point realization need to be improved. Inmetro decided to purchase zinc with 6N of purity in order to construct a new open zinc cell. The new cell will be tested in 2004-2005. Afterwards, a new tin cell will probably be manufactured too.

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