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PERFORMANCE OF A NEW SET OF IN, SN AND ZN TEMPERATURE FIXED-POINTS

A comparison of a new set of "In, Sn and Zn" temperature fixed-points on the International Temperature Scale of 1990 (ITS-90) was carried out at the National Institute of Standards (NIS – Egypt). The new set was manufactured by Hart Scientific – USA. The comparison was performed to measure their performance and capabilities in calibrating Standard Platinum Resistance Thermometers (SPRTs). This comparison was carried out using four calibrated SPRTs and the reference set of "In, Sn and Zn". The used reference set was manufactured by National Physical Laboratory (NPL-England). Full description and measurements made to the two sets are presented. Measurements showed good results and some agreement between the two sets. The differences between cells of the new set and reference cells were well within 2 mK. This showed their capabilities and the possibility of using them in calibrating SPRTs.

Keywords: ITS-90, SPRT, Fixed-Point

1. INTRODUCTION

The International Temperature Scale (ITS-90) specifies for the calibration of the SPRTs, the freezing point of indium (156.5985°C), tin (231.928°C) and zinc (419.527°C) [1]. Work on these fixed points was started previously in 1982 [2]. Development plans have encouraged the thermometry group to continue and improve the realization uncertainty [3, 4].

In the present work a comparison of a new set of "In, Sn and Zn" fixed points (set2), manufactured by Hart Scientific – USA, was carried out with the laboratory reference cells (set1). The two sets of cells are sealed cells corresponding to the design described in Supplementary Information for the International Temperature Scale of 1990 [5], and containing substances of at least 99.9999% purity. Four stable, calibrated, long-stem SPRTs are used through the comparison.

2. DESCRIPTION OF IN, SN AND ZN CELLS

The reference cells (set1) of fixed-points are manufactured by NPL-England. They are large cells of purities of 99.9999%, cast under an inert atmosphere. Each cell crucible has an external length of 240 mm. The graphite crucible is sealed in a slightly longer silica outer tube, with an integral silica lining tube extending to the bottom of the graphite well. Initially, the cell is pumped out and heated until the contents of the crucible are molten, and then argon is admitted and the pressure adjusted to one atmosphere at the fixed-point temperature. The silica pumping tube at the top of the cell is then sealed off. The cells are mounted in an Inconel holder (460 mm high), closed at the bottom, with a thermometer guide tube assembly above as described elsewhere in [6].

The new set of fixed-points (set2) manufactured by Hart Scientific – USA are small cells of purities of 99.9999% and having 43 mm outer diameter, 8 mm well inner-diameter and 214 mm height. Figure 1 shows the structure of the fixed-point cell for both sets.

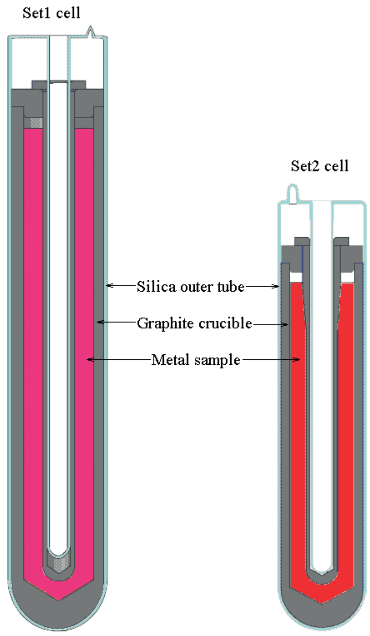

Fig. 1. Fixed-point cell.

Two "three-zone" furnaces are used to realize the fixed points. All measurements with SPRTs, described in Table 1, are done automatically using an AC automatic resistance bridge "ASL-F18", associated with 100 Ω "H. Tinsley" standard resistor maintained at 20°C and controlled by a computer.

Table 1. SPRTs specifications.

SPRT	Serial No.
H. Tinsley	274241
H. Tinsley	274242
H. Tinsley	274243
H. Tinsley	263585

Measurements were taken with one SPRT at set1 cell and set2 cell sequentially to determine the temperature difference between them. This difference can be determined directly by the change in the W value (R_t/R_{wtp}). The measurements were corrected for immersion depth and self-heating, but not for cell pressure because it is not possible to access the sealed cell pressure. Thus, the two sets of each fixed-point are characterized through two mutual temperature differences.

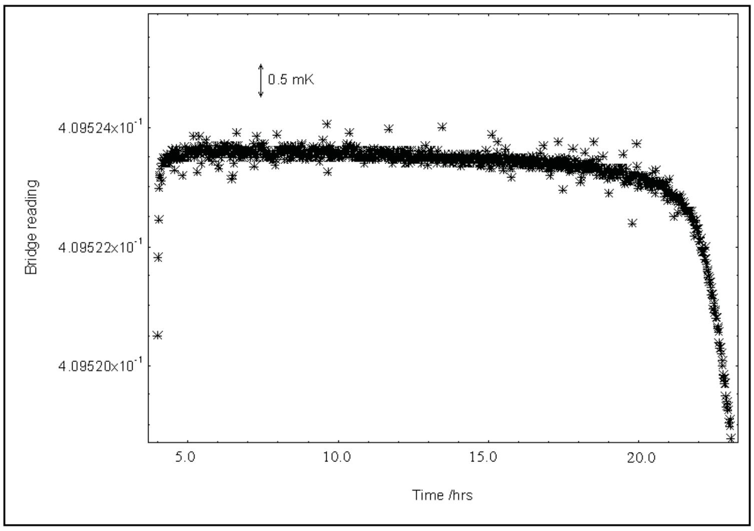

Fig. 2. A typical freezing curve of indium cell (set2).

The experiments were started by inserting the same fixed point cells from the two sets into the two furnaces using the same set point, such that the In cells were inserted first, and after finishing measurements the Sn cells were then replaced and finally the Zn cells were inserted. The set points used for the realization of the In, Sn and Zn freezing points were 156, 237, 425°C respectively the night before use, so that the ingot will melt overnight. In the morning the temperature is reduced to 153.6, 231, 419°C. Once a stable temperature on the plateau has been established, measurements were begun. A typical freezing, for example, of indium cell (set2) with these conditions is shown in Fig. 2.

3. RESULTS AND DISCUSSION

Since the temperature sensed by a thermometer in the cell will be higher than the freezing point due to the hydrostatic pressure effect, by an amount equal to 3.3, 2.2, 2.7 mK per meter of head in In, Sn, Zn respectively [1]. The depths in set1 and set2 cells are 17, 14.5 cm respectively, and the distance from the metal surface to the mid-point of the sensing element of the thermometer is about 14, 11.5 cm respectively. The head correction therefore amounts to 0.46, 0.30, 0.38 mK for In, Sn, Zn respectively of set1, and amounts to 0.38, 0.25, 0.31 mK for In, Sn, Zn respectively of set2. These are equivalent to 0.000045, 0.000028, 0.000032 Ω for the used thermometers, and may be corrected for by subtracting these values from the measured resistances. The measured resistances of the In, Sn and Zn cells were thus corrected for the self-heating effect and the hydrostatic head as shown in Table 2.

Table 2. Resistance to be measured with SPRTs of set1 and set2 fixed-point cells.

SPRT	274241	274242	274243	263585
In (set1)	--------	40.899623	40.954597	40.989607
In (set2)	--------	40.899476	40.954407	40.989416
Sn (set1)	47.725106	48.088775	48.153428	--------
Sn (set2)	47.724891	48.088569	48.153255	--------
Zn (set1)	64.770916	--------	65.352180	65.408287
Zn (set2)	64.770709	--------	65.352005	65.408035

Tables 3, 4 and 5 show the temperature differences found in In, Sn and Zn cells respectively in the two sets. The measured resistances of these tables are the averages of 15 runs for each SPRT.

Table 3. Measured differences of In cells.

SPRT	274242	274243	263585	Average
In (set1) /Ω	40.899623	40.954552	40.989562	--------
In (set2) /Ω	40.899476	40.954407	40.989416	--------
In cell diff. (set1-set2) /mK	1.46	1.45	1.46	1.46 ±0.01

Table 4. Measured differences of Sn cells.

SPRT	274241	274242	274243	Average
Sn (set1) /Ω	47.725106	48.088775	48.153428	--------
Sn (set2) /Ω	47.724891	48.088569	48.153255	--------
Sn cell diff. (set1-set2) /mK	2.14	2.06	1.73	1.98 ±0.22

Table 5. Measured differences of Zn cells.

SPRT	274241	274243	263585	Average
Zn (set1) /Ω	64.770916	65.35218	65.408287	--------
Zn (set2) /Ω	64.770709	65.352005	65.408035	--------
Zn cell diff. (set1-set2) /mK	2.07	1.75	2.52	2.11 ±0.39

The uncertainty components for the realization of all three points of set2 are

1) *Reproducibility* of the fixed point measured by the SPRTs,
2) *Plateau interpretation*, the more "probable" value is the one taken at the beginning of the plateau due to the thermal effect, nearly it is taken at 35% of solid melted,
3) *Electrical effects*, due to the bridge accuracy and short term stability of the standard resistor,
4) *Self heating*, due to the uncertainty of the contribution of the dispersion of the ratio between the two measuring currents, and of the bridge resolution,
5) *Hydrostatic pressure effect*, due to the uncertainty of the relative position of the middle of the thermometer sensing element and the metal liquid to solid phase,
6) *Impurities*, estimated through the chemical analysis given in the sample purity certificate,
7) *Spurious heat flux*, due to external heat fluxes came to the cell.

Table 6. Uncertainty budget of SPRTs calibration in fixed-point cells of set2.

Source of uncertainty	Uncertainty component (mK)		
	In	Sn	Zn
Reproducibility (A)	0.02	0.02	0.02
Plateau interpretation (A)	0.15	0.18	0.22
Electrical effects including F18 accuracy and standard resistor variation (B)	0.05	0.05	0.05
Self heating (B)	0.05	0.07	0.07
Hydrostatic pressure effect (B)	0.06	0.04	0.05
Impurities (B)	0.05	0.10	0.10
Spurious heat flux (B)	0.18	0.20	0.22
Combined uncertainty	0.26	0.30	0.34
Expanded uncertainty (k=2)	0.52	0.61	0.68

Table 6 shows the expanded uncertainties set2 fixed points. The uncertainty budget for set1 cells was previously described in [3, 4], their expanded uncertainties, $k = 2$, were 0.50, 0.61, 0.57 mK for In, Sn and Zn respectively.

4. CONCLUSION

The performance of the new fixed-point cells (set2) is similar to that of the reference cells, (set1) cells. The differences between the set2 and set1 cells were well within 2 mK. The expanded uncertainties of the set2 cells are almost the same as those applicable to the set1 cells. Thus, the set2 cells could be then used for most calibration purposes.

REFERENCES

1. Preston-Thomas H.: *The international temperature scale of 1990 (ITS-90)*. Metrologia, 27, pp. 3–10, 1990.
2. El-Shammaa H., Moussa M. R., Omar M. H.: *Realization of the triple point of water and the freezing points of tin and zinc at the National Institute of Standards (Egypt)*. Temperature its measurement and control in science and industry, vol. 5, pp. 205–208, 1982.
3. Ali K., Ahmed M. G., Moussa M. R., Rushdy M.: *Realization of Gallium and Indium fixed points on ITS-90 at National Institute for Standards (NIS) in Egypt*. MAPAN-Journal of Metrology Society of India, vol. 18, no. 2, pp. 51–56, 2003.
4. Ahmed M. G., Ali K.: *Realization of Tin and Zinc fixed points on ITS-90 at National Institute for Standards (NIS) in Egypt*. MAPAN-Journal of Metrology Society of India, vol. 19, no. 3, pp. 163–168, 2004.
5. Preston-Tomas H., Bloembergen P., Quinn T. J.: *Supplementary Information for the International Temperature Scale of 1990*. BIPM, Sevre, 1990.
6. Gamal M., Ali K., Moussa M. R.: *Realization of the ITS-90 over the Temperature Range 83 K to 693 K at the National Institute for Standards NIS-Egypt*. Temperature, Its Measurement and Control in Science and Industry, Symposium, New York, vol. 7, no. 1, pp. 59–64, 2003.