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STUDY OF SOME METAL FIXED POINTS FROM DIFFERENT SOURCES

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Abstract: A comparison of some defining temperature fixed-points on the ITS-90 was carried out at the National Institute of Standards using four calibrated SPRTs. The comparison was performed using large and small sealed cells from two different sources. The large cells, namely set1 cells and taken as reference cells were realized using the same technique used with the small cells (set2). The main task is to study the set2 cells and the possibility of using them instead of the large size (reference) cells. Measurements showed good results and some agreement between the two sets. The differences between the set2 cells and reference cells set1 were well within 2 mK.

Keywords: ITS-90, SPRT, Fixed-Point.

1. INTRODUCTION

One of the fundamental responsibilities of the National Institute of Standards (NIS) in Egypt is to establish, maintain and disseminate the International Temperature Scale of 1990 (ITS-90) [1]. To meet this task the thermal metrology laboratory of NIS has realized the ITS-90 fixed points from 83 K up to 2200 °C [2-5]. The metrological equivalence with the thermometry basis is established through inter-comparisons. In-between these inter-comparison, the stability of the individual fixed-point is verified by comparing two sets, each of three fixed points "indium (In), tin (Sn) & zinc (Zn)". The two sets are manufactured by two different sources: National Physical Laboratory (NPL-England "set1") and (Hart Scientific - USA "set2"). The two sets of cells are sealed cells, correspond to the design described in Supplementary Information for the International Temperature Scale of 1990 [6], contain substances of at least 99.9999 % purity and are of both design short and long. Four stable, calibrated, long-stem Standard Platinum Resistance Thermometers (SPRTs) are used through the comparison.

2. EQUIPMENT AND METHODS

The first set of fixed-points (set1), adopted as the reference cells, manufactured by NPL-England are large cells of purities of 99.9999 %. They have been cast under an inert atmosphere. Each crucible (external length of 240 mm) is then sealed in a silica outer tube, slightly longer than the graphite crucible, 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 [3].

The second 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 the both sets.

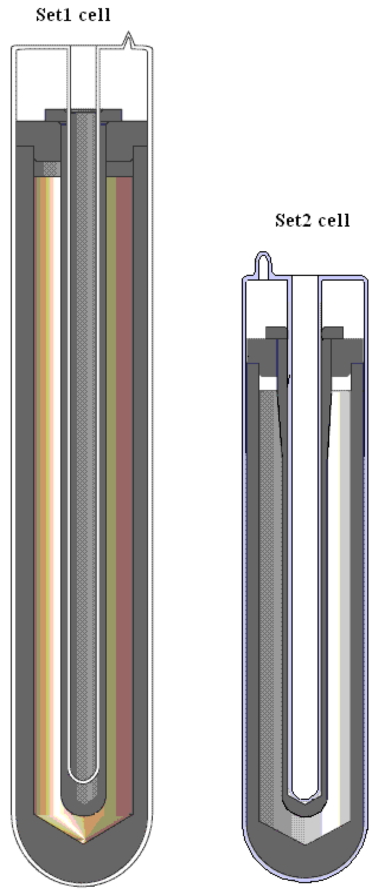

Fig. 1. Fixed-point cell

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, 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 a 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.

The experiments were started by inserting the same fixed point cells from the two sets into the two furnaces at each 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 melted overnight. In the morning the temperatures were reduced to 153.6, 231, 419 °C. Once a stable temperature on the plateau has been established, measurements were begun.

3. RESULTS AND DISCUSSION

A typical freezing, for example, of indium cell (set2) with the conditions described above is shown in Figure (2).

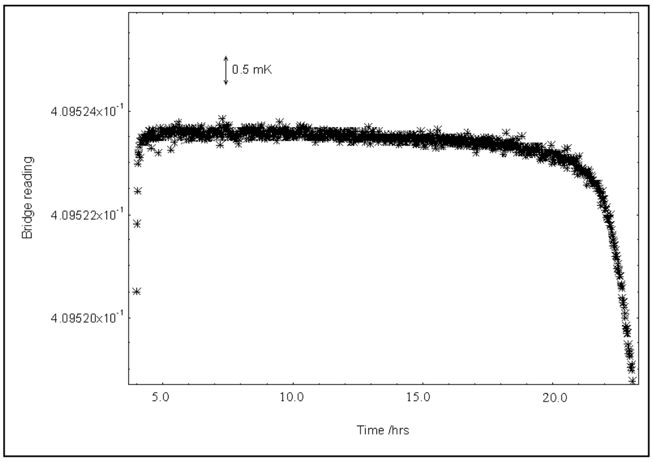

Fig. 2. A typical freezing curve of indium cell (set2)

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. 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. Measured resistances of fixed points.

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. Values in 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 slightly the mid of the plateau, nearly it is taken at 40% of solid melted, 3) *Electrical effects,* due to the bridge "linearity, symmetry, short term stability of the standard resistor", 4) *Self heating,* due to the uncertainty on the ratio between the two squared measuring currents, 5) *Hydrostatic pressure effect,* due to the uncertainty on 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) shows the expanded uncertainties of set2 fixed points. The uncertainty budget for set1 cells was previously described in [4,5], their expanded uncertainties, *k=2,* were 0.50, 0.61, 0.57 mK for In, Sn and Zn respectively.

Table 6. Uncertainty budget of set2 fixed points

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***

4. CONCLUSION

The performance of the fixed-point cells (set2) is similar to that of the set1 cells. The differences between the set2 cells and reference cells, set1, 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.

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