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Comparison of the Realizations of the ITS-90 over the Range from 83.8058 K to 692.677 K

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Abstract. In this paper the interlaboratory comparison in the field of measurement of temperature is presented. Within intercomparison calibration of a SPRT at fixed points in the range from the triple point of argon 83.8058 K (-189.3442 °C) to the freezing point of zinc 692.677 K (419.527 °C) was performed. The interlaboratory comparison was organized by the University of Ljubljana, Faculty of Electrical Engineering, Laboratory of Metrology and Quality (MIRS/UL-FE/LMK). The circulating items were two 25 Ω standard platinum resistance (SPRT) thermometers, one metal sheathed and one quartz sheathed. The interlaboratory comparison included maximum of six fixed points of ITS-90 in the range between -190 °C and 420 °C. However, certain laboratories didn't have all the fixed points in the range. They have performed only measurements in the fixed points that they have. Prior to the start of measurements, in case that the laboratory had the freezing point of zinc, annealing was performed as in accordance with protocol. MIRS/UL-FE/LMK performed measurements at the beginning of the interlaboratory comparison and at the end. Prior to the calibration at fixed points in each laboratory, test measurement at the TPW was done in order to assess stability of the instruments.

Keywords: standard platinum resistance thermometer, interlaboratory comparison, realization of ITS-90.

INTRODUCTION

The purpose of the interlaboratory comparison was to compare the results of the participating laboratories during calibration of a SPRT at fixed points in the range from the triple point of argon (-189.3442 °C) to the freezing point of zinc (419.527 °C). The circulating items were two standard platinum resistance (SPRT) thermometers, one metal sheathed and one quartz sheathed. The interlaboratory comparison included maximum of six fixed points of ITS-90 in the range between -190 °C and 420 °C. However, certain laboratories didn't have all the fixed points in the range. They have performed only measurements in the fixed points that they had. The protocol of intercomparison closely followed protocol of EUROMET 552 project (Key Comparison EUROMET.T-K3), which was registered in The International Bureau of Weights and Measures (BIPM) Key and supplementary comparisons (Appendix B) as EURAMET.T-K3, [1] and [2].

The interlaboratory comparison was part of the IPA 2008 proficiency testing project contracted by CEN/CENELEC and EFTA. Beside pilot MIRS/UL-FE/LMK (Slovenia), there were four other participants from the European region of Western Balkan and Turkey.

From Turkey participating laboratory was TÜBİTAK Ulusal Metroloji Enstitüsü (TÜBİTAK UME). The TÜBİTAK UME is Turkish national metrology laboratory, established in 1992. TÜBİTAK UME Temperature Standards Laboratory meets the needs of Turkish industry and scientific community for temperature calibrations and measurements in the range from -200 °C to 2600 °C by establishing the national temperature standards traceable to International Temperature Scale, ITS-90. Primary goal of the laboratory is the establishment of the national temperature scale and transfer of the scale to secondary laboratories. The Laboratory participated in number of international comparisons within EURAMET to prove the equivalence of the national scale.

From FYR Macedonia participating laboratory was Bureau of Metrology (BoM). Bureau of Metrology of the Republic of Macedonia is founded according to the Law on Metrology in 2002. Temperature laboratory has the equipment, which in primary temperature laboratory provides traceability directly to the International Temperature Scale ITS-90. Other

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equipment is used for dissemination of temperature by comparison calibrations.

From Republic of Serbia participating laboratory was Directorate of Measures and Precious Metals (DMDM). Directorate of Measures and Precious Metals develops, realizes, keeps, maintains and continuously improves measurement standards of the Republic of Serbia and provides their traceability to international level, provides metrological traceability, carries out metrological supervision, and conformity assessments of measuring instruments, performs other activities in the field of metrology in accordance with the Law. Group for thermometry covers main activities dealing with thermometry, humidity and heat energy.

From Croatia participating laboratory was Croatian Metrology Institute, Faculty of Mechanical Engineering and Naval Architecture, Laboratory for Process Measurements (HMI/FSB-LPM).

Number of above mentioned laboratories recently got, through different European projects and programs such as Community Assistance for Reconstruction, Development and Stabilization (CARDS and the instrument for pre-accession assistance (IPA), new and modern equipment. In order to include this new equipment and to confirm calibration and measurement capabilities, laboratories started intensively to participate in interlaboratory comparisons.

Interlaboratory comparison protocol

Prior to the start of measurements, in case that the laboratory had the freezing point of zinc, annealing was performed. The SPRT was carefully inserted into an annealing furnace at 470 °C, and then annealed for two hours at 470 °C. After thermal treatment, the SPRT was carefully removed from the annealing furnace directly to the room environment. The resistance value at the triple point of water was measured. If the resistance at triple point of water was increasing, the pilot laboratory had to be contacted immediately. If the decrease in the triple point of water resistance of the SPRT after annealing was equivalent to 0.5 mK or larger, the annealing procedure was repeated. If the decrease is less than 0.5 mK laboratory continued with measurements at fixed points.

If the decrease in the triple point of water resistance of the SPRT after second annealing was larger than 0.2 mK, the pilot laboratory was contacted for further instructions. Otherwise, laboratory continued with measurements at fixed points.

After the annealing, the SPRT was calibrated at all of the fixed points in the range of comparison, i.e., measurements at TPW, Zn, TPW, Sn, TPW, In, TPW, Ga, TPW, Hg, TPW, Ar and TPW, in that order. If one or several fixed points were not available then the host laboratory performed the comparison over a limited range. Existing techniques as used by the participating laboratory were used.

In order to not increase the uncertainty on the comparison of the results the R_T values given by the different participants approximately corresponded to the same percentage of metal in liquid phase, as described in the protocol of comparison, [4].

For each metal fixed point the $W=R_T/R_{TPW}$ was calculated. R_{TPW} is the TPW resistance measured immediately after the measurement of R_T. All the measurements at the fixed points had been corrected for self-heating, hydrostatic head and, if any, the pressure effect. At least 3 different phase transitions (3 freezing for Zn, Sn, In, 3 melting for Ga, 3 triple points for Hg and Ar) were performed. All three measurements for each fixed point were reported in the Excel spreadsheet including the calculated mean.

MIRS/UL-FE/LMK performed measurements at the beginning of the interlaboratory comparison and at the end. Prior to the calibration at fixed points in each laboratory, test measurement at the TPW was done in order to assess stability of the instruments.

In the report form, the participants were also asked to fill in details about the applied method, uncertainty sources, equipment and traceability. Measurements were performed from March 2010 until November 2010. Depending on the number of fixed point cells at which measurements were performed, each laboratory had from two weeks (for one cell) up to six weeks (for six cells).

STABILITY MEASUREMENTS

Prior to the start of the measurements at the fixed points, each laboratory had to perform measurements at the triple point of water. This measurement was used us check to see if something went wrong with SPRT as a result of transport between participating laboratories. All the measurements were corrected for hydrostatic head and self-heating.

While one thermometer with quartz sheath was stable and within expected limits, other thermometer with metal sheath at certain point in time become unstable and changed its triple point of water resistance value by more than 40 mK, Figure 1.

Prior to the interlaboratory comparison, test measurements at the triple point of water were done in order to assess stability of the instruments. Summary of the results for unstable thermometer can be seen on the Figure 2.

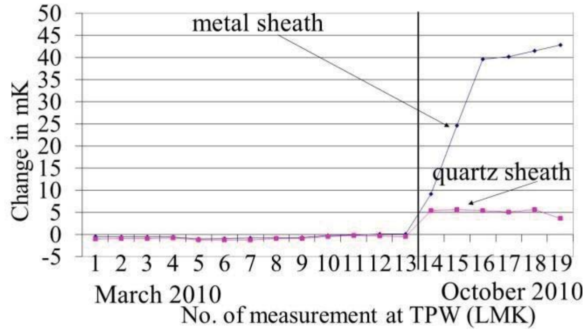

FIGURE 1. Behavior of resistance of both SPRTs at the triple point of water

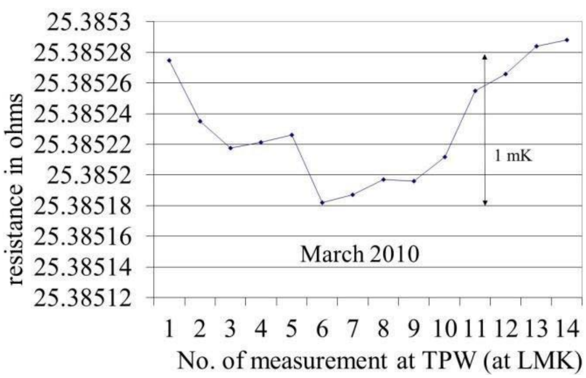

FIGURE 2. Stability of metal sheathed SPRT before interlaboratory comparison.

As it can be seen from Figure 2, the thermometer with metal sheath was stable prior to the start of interlaboratory comparison. There was no prior indication that it could become unstable during the intercomparison.

It is not clear what caused this significant change. After contacting manufacturer and consulting literature we found out that the usual cause of increasing R_{TPW} is the depletion of available oxygen within the sheath, [3]. Once this occurs, the SPRT is usually of no use. As a consequence, we have used only results of quartz sheathed SPRT in this interlaboratory comparison.

The change of *W* value at other fixed points for the quartz sheathed thermometer, from first to last calibration at MIRS/UL-FE/LMK can be seen at Figure 3. This change was taken into account as uncertainty contribution to the uncertainty of the reference values at each fixed point.

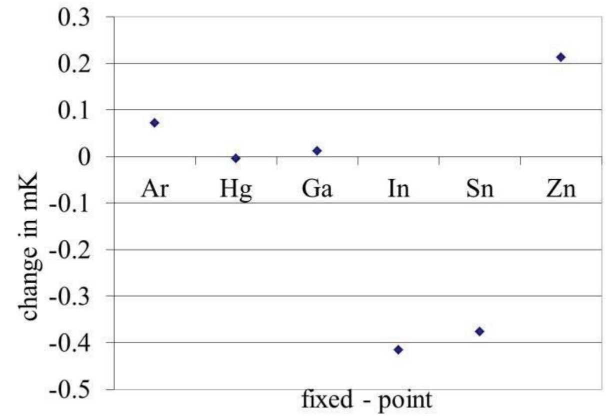

FIGURE 3. Change of *W* value of quartz sheathed SPRT at other fixed points.

RESULTS AND UNCERTAINTIES

Table 1 present the *W* values given by the participants at the different fixed points together with their uncertainties in mK.

From the literature, such as report for CCT-K3 and EUROMET T.-K3(Project 552), [1 and 2], reference values have been determined as mean, weighted mean (with uncertainty used as weight) and median. In one case, reference value was determined as average of the mean, weighted mean and median. Due to relatively small number of participants (in case of argon point only two and in case of mercury five), we have decided to calculate only weighted mean. So, as a reference value at each fixed point, measurement values with their corresponding uncertainties measured by the all participants during the course of the interlaboratory comparison were taken into account.

The resulting reference values were taken as the weighted mean value from those measurements, using uncertainties from each laboratory as weight.

$$W_{wm} = \frac{\sum_{i=1}^{n} \frac{Wlab_i}{u_i^2}}{\sum_{i=1}^{n} \frac{1}{u_i^2}} \qquad (1)$$

where W_i is *W* value as determined by each laboratory at each fixed point and u_i is uncertainty of that value. The uncertainty of the reference value W_{wm}, u_{Wwm} was calculated as:

$$u_{W_{wm}} = \sqrt{\frac{1}{\sum_{i=1}^{n} \frac{1}{u_i^2}}} \qquad (2)$$

On the figures 4 to 9 one can see difference of the participants from the reference value together with uncertainty of reference value (dashed lines).

TABLE 1. Measurements and uncertainties during the calibration of quartz sheath SPRT

Fixed point	*W*(MIRS/UL-FE/LMK)	*U*(MIRS/UL-FE/LMK) in mK	*W*(DMDM)	*U*(DMDM) in mK	*W*(TÜBİTAK UME)	*U*(TÜBİTAK UME) in mK
Ar	0.215884943	0.8			0.215886040	0.98
Hg	0.844146382	0.6	0.844146931	0.69	0.844147892	0.39
Ga	1.118132856	0.4	1.118134641	0.84	1.118132111	0.52
In	1.609761346	1.2	1.609763513	1.9	1.609762345	1.67
Sn	1.892737927	1.0	1.892741254	1.59	1.892740553	1.11
Zn	2.568803568	1.5	2.568806592	1.94	2.568805453	1.49

Fixed point	*W*(HMI/FSB-LPM)	*U*(HMI/FSB-LPM) in mK	*W*(BOM)	*U*(BOM) in mK
Ar				
Hg	0.844145648	1.36	0.84414109	1.32
Ga	1.118132627	0.96		
In				
Sn	1.892739477	2.4		
Zn	2.568797289	3.29		

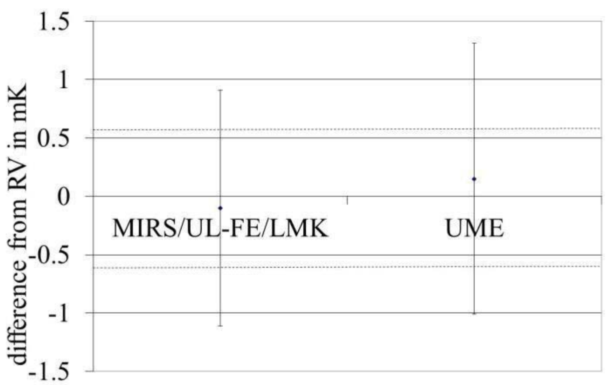

FIGURE 4. Differences at Ar triple point.

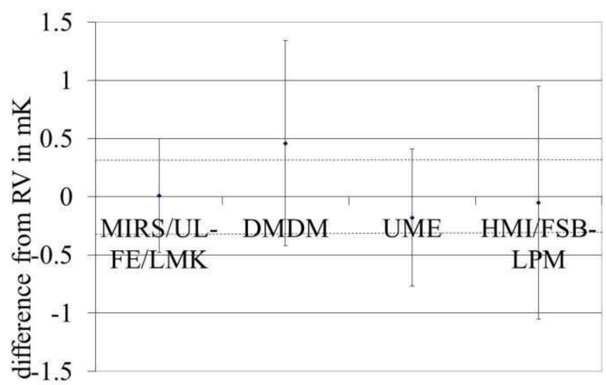

FIGURE 6. Differences at Ga melting point.

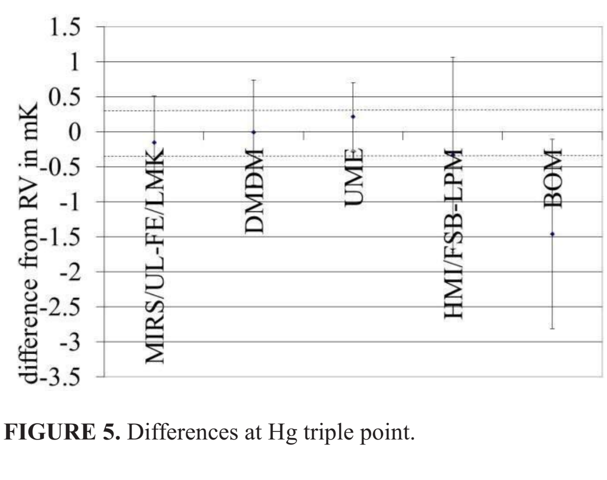

FIGURE 5. Differences at Hg triple point.

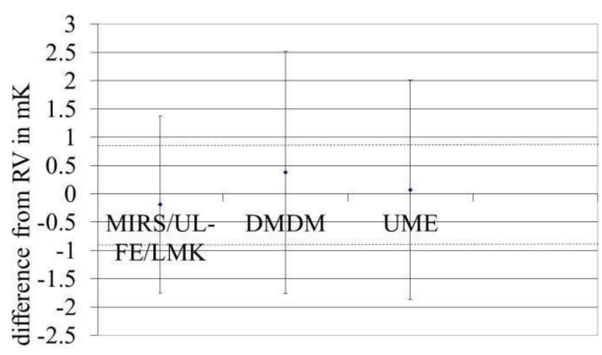

FIGURE 7. Differences at In freezing point.

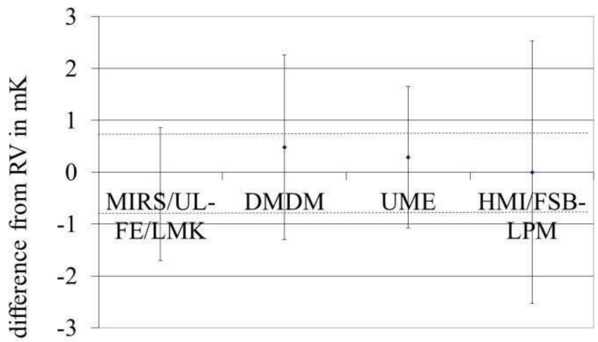

FIGURE 8. Differences at Sn freezing point.

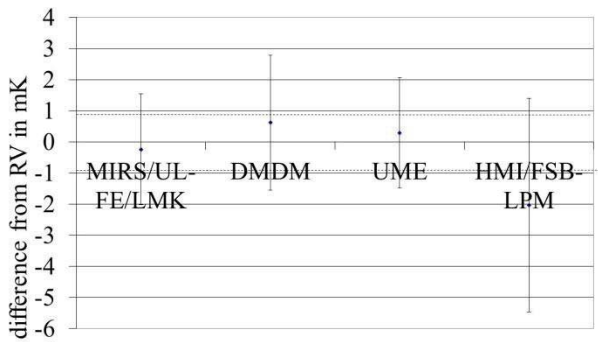

FIGURE 9. Differences at Zn freezing point.

Uncertainties

Each participating laboratory provided resistance ratios, measured in their local fixed points. Each participant was requested to supply the uncertainty budget associated with the calibration at the different fixed points.

In Table 2 there is a summary of uncertainty sources for the calibration of SPRT at the zinc fixed point, as an example of uncertainty budget in calibration of SPRTs by fixed point method.

For the components estimated using a type B method the degrees of freedom are assumed to be infinite. For the component estimated using a type A method, the degrees of freedom depends on the number of results. The combined uncertainties were computed by root-sum-of-squares of the type A and type B contributions.

Whatever the fixed point and the laboratory considered, the coverage factor t_{95}(n) from the *t*-distribution is very close to 2. So, for all the laboratories coverage factor *k=2* was used in order to calculate the combined expanded uncertainties.

In addition to the uncertainties reported by each laboratory an uncertainty, for possible instabilities of the circulated SPRT over the course of the comparison, has to be taken into account. Furthermore, uncertainty due to reproducibility of the MIRS/UL-FE/LMK calibration at fixed points represent additional source of uncertainty.

Both uncertainties were taken as rectangular distribution. The stability of SPRT was calculated as difference between first and last calibration at MIRS/UL-FE/LMK

$$u_{StabSPRT} = \frac{\left|W_{FP_{end}} - W_{FP_{begin}}\right|}{\sqrt{3}} \times \left(\frac{\delta T}{\delta W}\right)_T \qquad (3)$$

The reproducibility of the MIRS/UL-FE/LMK calibration at fixed points is calculated from number of calibrations of same thermometer at fixed point:

$$u_{reproducibility} = \frac{\left|W_{1559\max} - W_{1559\min}\right|}{\sqrt{3}} \times \left(\frac{\delta T}{\delta W}\right)_T \qquad (4)$$

These uncertainties were taken into account as uncertainty contribution to the uncertainty of the reference values at each fixed point. As you can see from figure 3, they were significantly smaller than other uncertainty contributions.

CONCLUSION

The EURAMET 1167 project compares the various local realizations of the ITS-90 from the triple point of Ar (83.805 8 K) to the freezing point of Zinc (692.677 K) using long-stem SPRTs. Participating laboratories are national measurement laboratories in IPA countries (Former Yugoslav Republic of Macedonia, Croatia, Republic of Serbia and Turkey). This comparison is coordinated by MIRS/UL-FE/LMK, as Slovenian national laboratory.

The comparison was performed in one loop. In order to have sufficient information about a possible drift of the SPRTs, the coordinator has performed a calibration over the full temperature range at the beginning and at the end of the loop.

Given that the protocol of the comparison contains a detailed description of how the uncertainties are to be calculated, the uncertainty budgets established by the participants seem consistent or, at least, homogeneous.

The results of the comparison were analyzed by the coordinator. The reference value used was the weighted mean of the results at each fixed point.

We can conclude that all the measurements performed by the participants were within expected limits and uncertainties, as described in [1] and [2], as well as from acquired equipment data, [4].

ACKNOWLEDGEMENTS

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3. BIPM, Supplementary Information for the International Temperature Scale of 1990 (1997)
4. Project EURAMET 1167, draft A report, February 2012

TABLE 2. Uncertainty sources for the calibration of SPRT at the freezing point of zinc

Components	DMDM Uncertainty contribution u_i in mK	HMI/FSB-LPM Uncertainty contribution u_i in mK	TUBITAK TÜBİTAK UME Uncertainty contribution u_i in mK	MIRS/UL-FE/LMK Uncertainty contribution u_i in mK
Repeatability of readings	0.08	0.007	0.02	0.03
Uncertainty linked with purity	0.7	0.5	0.7	0.4
Uncertainty linked Hydrostatic pressure correction	0.005	0.054	0.03	0.012
Uncertainty linked with perturbing heat exchanges	0.1	0.2	0.17	0.1
Uncertainty linked with self-heating correction	0.2	0.03	0.14	0.03
Uncertainty linked with bridge linearity	0.11	0.083	0.04	0.05
Uncertainty linked with AC/DC current	0	0.03	0.01	0
Uncertainty linked with gas pressure	0.04	0.020	0	0.05
Repeatability of readings	0.04	0.007	0.012	0.02
Repeatability of temperature realized by cell	0.3	0.088		0.05
Short repeatability of calibrated SPRT	0.32	0.17	0.017	0.15
Uncertainty linked with purity and isotopic composition	0.30	0.09	0.041	0.05
Uncertainty linked Hydrostatic pressure correction	0.005	0.05	0.005	0.005
Uncertainty linked with perturbing heat exchanges	0.12	0.09	0.02	0.01
Uncertainty linked with self-heating correction	0.15	0.09	0.06	0.03
Uncertainty linked with bridge linearity	0.27	0.22	0.02	0.05
Uncertainty linked with AC/DC current	0	0.088	0.01	0
Uncertainty linked with internal insulation leakage	0.004	0.088	0.002	0
Uncertainty linked with stability of R_S	0.008	0.008	0.01	0
Uncertainty linked with temperature of R_S	0.008	1.47	0.05	0.005
Wt scatter	0.02	0.35	0.05	0.59
Combined uncertainty	**0.975**	**1.65**	**0.75**	**0.75**
Expanded uncertainty	**1.95**	**3.3**	**1.50**	**1.50**
k=2				