XVI IMEKO World Congress
Measurement - Supports Science - Improves Technology - Protects Environment ... and Provides Employment - Now and in the Future
Vienna, AUSTRIA, 2000, September 25-28

THE ITS-90 DISSEMINATION THROUGHOUT BRAZIL

D.M. Camarano
Thermal Metrology Division
National Institute of Metrology, Standardization and Industrial Quality of Brazil – INMETRO; Rio de Janeiro, Brazil

Abstract: This paper describes the realization of the International Temperature Scale of 1990 (ITS-90) from 83,8058 K to 692,677 K at INMETRO. A brief review is given of the apparatus employed at the thermometry laboratory. Additionally, it also shown how the uncertainties are evaluated following closely the rules prescribed in the ISO guide to the Expression of Uncertainty in Measurement.
Keywords:Thermometry,ITS-90, Uncertainty of Measurement.

1 INTRODUCTION

The National Institute of Metrology, Standardization and Industrial Quality of Brazil (INMETRO) is responsible for realizing, maintaining and disseminating the International Temperature Scale of 1990 [1] for Brazil. The calibration of standard platinum resistance thermometers (SPRTs) and high temperature platinum resistance thermometers (HTPRTs) at the defining fixed points is one of the methods used for the dissemination of the scale. The bulk of INMETRO experimental activity is concentrated in the medium temperature range between the triple point of Argon (83,8058 K) and the zinc freezing point (692,677K). Additionaly, a brief description of some quality techniques used in order to minimize and characterize the sources of uncertainties associated with standard platinum resistance thermometers calibration is presented, such as monitoring of the realization of fixed points cell, daily check up of the measurement system using reference resistors, measurement of excitation current, comparison of curves shapes of melting and freezing points for each fixed point, direct comparason of cells of the same fixed point, stability and uniformity of temperature of furnace used for the realization of fixed points.

2 REALIZATION OF THE ITS-90

INMETRO keeps a set of fixed points cells that are undergo a continuing check. The frequent monitoring of the realization of each fixed points can indicate a problem at the fixed point, or in the procedure used in laboratory. Several furnaces and other environmetal chambers are used to the realization of each fixed point, such as cryostats and furnaces. Standard long-steam platinum resistance thermometers (SPRTs), Tinsley and L&N are used as interpolating thermometers between the argon triple point up to zinc freezing. The thermometer resistance measurements are made with two the Precision Thermometry Bridges, model F-18 (ASL) and model 9975 (Guildline), and Tinsley standard resistor placed into a bath with controlled temperature.The monitoring thermometers is measured only at its defining fixed point and a triple point of water to determine the resistance ratio $W(T_{90})$, $W = R/R_w$.The reproducibilities obtained for monitoring thermometers during the realization of the fixed points are used in the determination of the uncertainty of a thermometer calibration (Table 1).

Table 1. INMETRO fixed points

Fixed points	State	Number of fixed points realizations	Standard deviation (1σ) / mK
Argon	triple point	8	0,40
Mercury	triple point	6	0,16
Gallium	melting point	13	0,15
Indium	freezing point	6	0,30
Tin	feezing point	6	0,29
Zinc	freezing point	10	0,30

3 UNCERTAINTY OF MEASUREMENT

For each type of calibration, the contribuitions of the various uncertainties are combined to give the total uncertainty. The model used at INMETRO follows closely the principles in ISO guide to the Expression of Uncertainty in Measurement [2]. In this model used at INMETRO [3], all conceivable sources of uncertainty and their correlations are taken into account. Several sources of uncertainties had been examined in order to identify their influence in the final thermometer calibration. Others laboratories use similar approach, but it is clear that the value and the method of analysis of each individual source of uncertainty vary among the latoratories. This is in large part due to the difficult to classify and measure some of the factors contributing to the final uncertainty. Not only the laboratories need to identify the sources of uncertainty, but also to implement procedure to guarantee the quality of measurement.

3.1 Resistance bridge and standard resistor

The accuracy of the resistance measurement depends on the accuracy of the bridge and the stability and accuracy of the standard resistor. At INMETRO, one of the methods for testing the bridge is the use of reference resistors (e.g. 1 Ω, 10 Ω, 25 Ω and 100 Ω). The measured ratio between two reference resistors (R1/R2) should not exceed the measurement uncertainty resulting from that of the bridge and that of the calibrations of the resistors. Other test used in the laboratory consists in interchange the ratio (R2/R1). The second measured ratio should agree with the first at about ± 0,4 ppm. Furthemore, checks are performed daily on the measurement system before any measurement. Three reference resistors daily are intercompared to verify the measurement system. The total change does not exceed ± 0,05 mK.

The stability of the standard resistor is dependent on the control of the resistor temperature. The thermal oscillations of the bath are accounted for the deviations of standard temperature from its nominal value (T_o) and resistance coefficient (γ) supplied by the manufacturer of the standard resistor. The total deviation of temperature of the standard resistor does exceed 0,01 °C. The uncertainty of γ is not given by the manufacturer but can be obtained experimentely. In most cases, in the calculation of uncertainty of measurement, its coefficient of sensitivity vanishes.

The thermometer resistance measurements are made with two Precision Thermometry Bridges, AC model F-18 (ASL) and DC model 9975 (Guildline). In most cases, the difference between the two bridges does not exceed 0,15 mK.

Resistance bridge measure resistance as a dimensionless ratio *r* (average of the bridge indications), of the thermometer resistance *R'* to a standard resistance R_s.The random effects of the bridge, such as linearity and current stability, are taken into account by introducing a correction factor ξ, nominally equal to one. Thus the thermometer resistance *R*' is then modeled as

$$R' = r \cdot x \cdot [1 + g\,(T_s - T_o)] \qquad (1)$$

where T_s is the actual temperature fo the standard resistor. For the realization of each fixed point the quantities T_s and ξ varied independently. The uncertainty of ξ is supplied by manufacturer of the bridge. The uncertainty of *r* is estimated to be the calculated standard deviation obtained from ratio measurements. For Type B uncertainty of T_s is used U-shaped probability distribution and for ξ is used rectangular probability distribution.

Variations in the excitation currents of the resistance bridges are verified using reference resistors and a digital multimeter. The total change of the measuring current of the ac bridge does not exceed 0,08 % for the 1mA and 1,4 mA. At moment for the uncertainty of others currents is used values obtained from technical literature pertaining to the bridge. Measurements of the SPRT resistance are done at two measuring currents so that the resistance value could be calculated for null-power dissipation. Datas for null-power dissipation are used to eliminate error from self-heating of the thermometer, the extrapolated resistance for zero current is calculated as

$$R_0' = R_1' - \frac{I_1^2}{I_2^2 - I_1^2} \cdot (R_2' - R_1') \qquad (2)$$

where I_1 and I_2 are the two sensing currents.

3.2 Fixed points

Hart Scientific cells for the triple point of water and one from CENAM (Centro Nacional de Metrología – Mexico) are available at INMETRO. For the realization of Zinc, Tin and Indium fixed points are used sealed cells manufactured by Engelhard Pyro-Controle. For the triple point of argon is used a cell manufactured by SORIME, under license by INM-France (National Institute of Metrology). For the melting point of gallium and triple point of mercury are used cells built by Isotech. The purity of the fixed points substances is usually 6N. As part of an internal measurement control, the cells used as laboratory standards are periodically compared to ensure that they have not changed with time. We verified a cell in the following ways: by melt curve slope, by freeze slope and the slope of 50 % of the freeze, by the difference between melt and freeze temperature,and plateau duration. For our cells, the slope of 50 % freeze/melt curves are less than 0,5 mK for Zn; 0,3 mK for Sn; 0,2 mK for In and 0,07 mK for Ga. For the triple point cells Ar and Hg, the plateau is flat within 0,1 mK and 0,2 mK, respectivelly. Moreover, all curves are compared with these obtained previously.

To obtain the corrected resistande R at the ITS-90 temperature T, corrected for hydorstatic pressure head, it is assumed that:

$$R_{\mathrm{W}} = \frac{R'_{\mathrm{W}}}{1 + (T'_{\mathrm{W}} - T_{\mathrm{W}}) dW_{\mathrm{r}} / \mathrm{d}T \big|_{T'_{W}}} \tag{3}$$

and

$$R_{a} = R'_{a} + (T_{a} - T'_{a}) R_{\mathrm{W}a} \frac{\mathrm{d}W_{\mathrm{r}}}{\mathrm{d}T \big|_{T'_{a}}} \tag{4}$$

The subscript ∝ is used to represent all fixed points in any given subrange, excluding that of water where is used W. The temperatures at these points of measurement are obtained from $T' = T_{\mathrm{c}} + Bh$, where T_{c} is the temperature realized in the cell, obtained from comparison measurements, B is the immersion coeffficient and h is the hydrostatic head.

The uncertainty in the immersion depth due to the uncertainty in the position of the SPRT sensor (h) is estimated to be 3 mm. The uncertainty of B, immersion coefficient, is assumed maximum possible deviation of the actual value of the quantity from its best estimated value. For Type B uncertainties of h and B is used retangular probability distribution.

3.3 Furnaces/criostats

Two furnaces of three-zone are used to operate from Zinc fixed point to Indium fixed point. These furnaces have a main heater with additional heaters controlled by separate controllers at each end to minimize heat losses out of the ends. The temperature of each furnace is controlled to within ± 0,03 mK, and the temperature profile along thermometer wells of the cells measured at temperatures close to the freezing points, was found to be uniform to within 0,2 K over the entire 180 mm length fo the metal ingots.

The triple point of argon is realised in a cryostat made by the SORIME. For the mercury triple point and melting point of gallium realisation it is used an apparatus a commercial Isotech equipment.

With the described equipments, it is possible to achieve plateau for the freezing points of zinc, tin and indium longer than 20 hours. For the triple point of argon, the plateau is within 4 hours. For the triple point of mercury and melting point of gallium, plateau lasting more than 18 hours are obtained.

3.4 Mathematics of ITS-90

According to the ITS-90 definitions, the temperature is calculated from reistance measurements. A reference function and the relation giving the difference between the resistance ratio, $W(T_{90})$, of the calibrated thermometer and that of the ITS reference one are then used to deduce the temperature between the calibration points (deviation function).

To obtain the uncertainty reported in the calibration certificate, the temperature is expressed as function of coefficients of the deviation function specific of each range. For example, a SPRT calibration at the water, tin and zinc points, we can estimate the combined standard uncertainty (u_c) in the measured temperature as

$$u^2{}_c(T) = \left(\frac{\partial f}{\partial a}\right)^2 u^2(a) + \left(\frac{\partial f}{\partial b}\right)^2 u^2(b) + 2\left(\frac{\partial f}{\partial a}\right)\left(\frac{\partial f}{\partial b}\right) u(a,b). \tag{5}$$

Where *a* and *b* are coefficients of the deviation function obtained from measurements at the defining fixed points.

Additionally, INMETRO staff has studied the inherent uncertainties of the ITS-90 such as the dispersion due to the so-called non-uniqueness of the scale. For the ITS-90, there are tree types of non-uniqueness [4]. The first type arie from the application of different equations in overlapping ranges, using the same thermometer. The econd type arises from the use of different kinds of thermometers in overlapping ranges. The third type of non-uniqueness arises from the characteristics of the standard thermometer. The non-uniqueness due to application of different equations in overlapping ranges, using the same thermometer are, in most cases, quite acceptable. Only the experiment involving the sub-range 273,16 K to 691,677 K relative to sub-range 273,16 K to 429,7485 K indicates a value that is larger than expected (1,6 mK). The non-uniqueness that arises from the characteristics of the standard thermometers at the fixed points of In and Ga are about ± 0,2 mK at both fixed points (at 273,16 K to 692,677 K).

Besides, as part of quality control of measurements, we use the analysis of the difference between W_{SPRT} and $W_r(T_{90})$ versus W_r obtained from a set of SPRTs. Thus, the relationship between W_r and any given W_{SPRT} may be useful to point out an experimental error such as systematic deviations of a particular fixed point. The expanded uncertainty of the fixed points realization at INMETRO are given in table 2.

Table 2. The uncertainty of the fixed point realization at INMETRO

Fixed Points	Uncertainty (*k* =2) / mK
Argon	1
Mercury	1
Water	0,1
Galliun	1
Indium	2
Tin	3
Zinc	4

The problem of uncertainty evaluation in fixed point comparisons represents another important aspect to be studied, moreover, the implementation of the ISO Guide to the Expression of Uncertainty in Measurement [2] should receive more attention. The process of intercomparison is subsequently performed in measurement of the difference of temperature between the fixed point cells. Temperature difference may be deduced from resistance (*R*) difference equation:

$$T_x - T_y = (R_x - R_y)\frac{dT}{dR} \qquad (6)$$

The resistance difference may be converted in temperature by means of the sensitivity at thermometer coefficient d*T*/d*R*, characteristic of the thermometer. Each resistance determination is obtained as described using the equations previously presented. At least, two thermometers are used and more accurate tests should be used, such as the classical tool of analysis of variance (ANOVA). Variations of thermometer characteristics, related to manipulation when passing from cell to cell, are assumed to be negligible. Besides, we use the gallium point as an alternative to check thermometer stability. The task in evaluating measurement consists in setting up the model function using the equation 6. However, the model used deserves further investigation with a thorough study of correlations.

CONCLUSIONS

A wide range of proceedings have been implemented that is some cases can diagnose faults in measurements. We have attempted to study several aspects of the uncertainty in fixed point measurements in order to improve the results. We observe that the main uncertainty component arises from cell uncertainty obtained from comparison measurements.

The results presented are acceptable for accurately disseminating the ITS-90 through calibration of SPRTs. International comparisions are an important part of maintaining brazilian national standards in measurement and we have plans that involve comparisons of fixed point cells.

REFERENCES

[1] H. Preston-Thomas H., The International Temperature Scale of 1990, *Metrologia* 27 (1990) 3-10.
[2] ISO, Guide do Expression of Uncertainty in Measurement , 1990.
[3] Lira I.,Camarano D.M., Villalobos J.P., Santiago. F., Expression of the Uncertainty of Measurement in Calibration of Thermometers. Part 1 : SPRTs, *Metrologia* 36 (1999) 107-111.
[4] Mangum B.W., Bloembergen P., Chattle M.V., Felmult B., Marcarino P., Pokhodun A.I., On The International Temperature Scale of 1990 (ITS-90), *Metrologia* 27 (1997) 3-10.

AUTHOR: Thermal Metrology Division (DICAL), Thermometry Laboratoy (LATER), Av. N. Senhora das Graças, 50, Duque de Caxias, Xerém, 25250-010, Rio de Janeiro, Brazil, Phone/FAX ++55 21 679 9027, E-mail: inmetro-mg@montreal.com.br