Inhomogeneity Influence In Thermocouple Calibrations

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ABSTRACT

In the International Temperature Scale of 1990 (ITS-90), any thermocouple figures out as an interpolation or a transfer measuring standard in its realisation. Yet, their use was largely disseminated in previous temperature scales. In daily work, National Metrology Institutes (NMIs) keep on calibrating some noble metal thermocouples to be used by accredited laboratories and industries. Mainly types R and S thermocouples are used as work standards, for calibration by comparison, in those laboratories which are in the lower levels of National Traceability chain. It is known that calibration depends on the depth in which the measuring instrument, thermocouple in this case, is settled into the furnace. Of course, we intend to consider for this hypothesis that different furnaces have a proper thermal distribution. But, how large is the influence of depth and, hence, non-homogeneity in the comparison of calibration results in different calibration set-ups?
Using fixed point cells in their changing phase soaks as ideal thermal means, Brazilian Thermometry Group, as the dissemination technology body in Temperature of Brazilian Accredited Laboratories for Calibration (RBC), presents some results on a study of non-homogeneity influence of types R and S thermocouples, reporting their behaviour when they are moved up or down in the ingots of cells. This characteristics of thermocouples, which have alloy wires, is strengthened when we compare their results with Gold-Platinum thermocouples and Standard Platinum Resistance Thermometers.
Another aim of this work is to discuss uncertainty evaluation for noble metal thermocouples. May Laboratories assure the reproducibility of too small uncertainties for thermocouples which have alloy wires? According to data acquired in this work this tends to have a limit, which is largely discussed by Brazilian Thermometry Group.

1. INTRODUCTION

Electromotive force (emf) in a thermocouple depends essentially on the temperature gradient between measuring junction and reference junction . Although, inhomogeneities along wires tend to affect results, for impurities usually generate micro thermocouples in both elements. If those micro thermocouples are set in temperature gradient zone which is created between room temperature and the top zone of calibration thermal device, the voltmeter may be indicating a different emf in relation to the measuring junction emf.

The uncertainty component due to the inhomogeneities is difficult to be assigned in the combined uncertainty. In the Thermometry Laboratory of Inmetro, the fixed point calibration method for types R and S thermocouples, for example, the uncertainty budget does not compute this component. It is combined with the stability [1].
Emf stability is the main factor to describe the thermocouple reproducibility throughout lifetime. Wires contamination, magnetic effects, mechanical strain (cold work) and inhomogeneities are present during the use of standard thermocouples [2]. These factors modify the thermocouple calibration curves and the inhomogeneities may modify also along the wires during the use. Thus, it is not easy to estimate the value of the inhomogeneity uncertainty contribution.
This work evaluates inhomogeneities in types R, S, and Au/Pt thermocouples in fixed point cells. Some graphs are presented for different immersion positions in the cells and the temperatures of furnaces were changed to verify its influence in the measured emfs.

2. FACILITIES.

In this work, thermocouple inhomogeneities were evaluated during the fixed point calibration in two fixed points sealed cells: Silver and Aluminium. All substances are kept in fixed point cells - graphite crucibles maintained in inert atmosphere inside borosilicate or quartz containers. The substances in those cells are of very high purity (99,999%) which are used in sealed cells (manufactured by Isotech). A Potassium heat pipe furnace was used with the cells (High Temperature furnaced manufactured by Isotech).

The reference junctions of thermocouples were kept in dewar vases with ice produced from distilled water. Two types of voltmeters are used to measure thermocouple electromotive forces: Hewlett Packard DMM model 3457A and Keythley nanovoltmeter model 182. Both are automatically read by a personal computer which works with a Visual Basic data acquisition program developed in Inmetro.

3. THERMOCOUPLES AND INHOMOGENEITY

Since Seebeck formulated the thesis of thermal circuit, thermometry scientists discovered several features which influence thermal measurements using thermocouples. Seebeck thought electromotive force (emf) was settled as the result of temperature difference between measuring and reference junctions. Yet, it was discovered later that thermal gradient along wires is the actual responsible for emf generation. Moreover, it was studied that temperature causes degradation in thermocouple wires throughout their lifetime. The aim of this work is to demonstrate how far alloy degradation can induce different responses in thermocouple calibrations throughout their lifetime.

There are several alloys employed in thermocouple manufacturing. In this paper, only three types of thermocouple alloys are going to be analysed (listed bellow), because they represent the most common standard thermocouples which are calibrated by Brazilian Thermometry Group and Brazilian Accredited Laboratories for Calibration (RBC).

- Type S: Platinum 10% Rhodium (+) / Platinum (-);

- Type R: Platinum 13% Rhodium (+) / Platinum (-);
- Gold (+) / Platinum (-).

It may be realised that the last thermocouple listed above is not made of an alloy, but from pure wires (99,999%). This is a path which thermometry scientists are following in the last years in order to decrease uncertainties due to calibrations and to make them much more trustful.

Inhomogeneity is a variation in chemical composition on the physical state of a substance. A thermocouple should be derived from materials of as high quality as is available, as closely related in position on a spool as possible, and the supply of thermocouple materials should be from one tested lot in a quantity adequate for the entire job[3].

Despite taking steps in order to prevent thermocouple degradation due to overheating, either during calibration by comparison or by fixed points, wires should have their chemical composition changed by ion migrations along wires which form thermal circuit, by evaporation of elements and by contaminants originated from the atmospheres they are calibrated. Annealing is pointed out as an ideal procedure to maintain thermodynamic equilibrium and to ensure metallurgical stability over the temperature range of application. Although, technicians should have in their minds that if the positive thermoelement of a type R thermocouple, for instance, decreases in 1% of Rhodium, there is no annealing procedure which may bring this thermocouple emf back to its previous status. This fact characterises emf drift of thermocouples throughout lifetime.

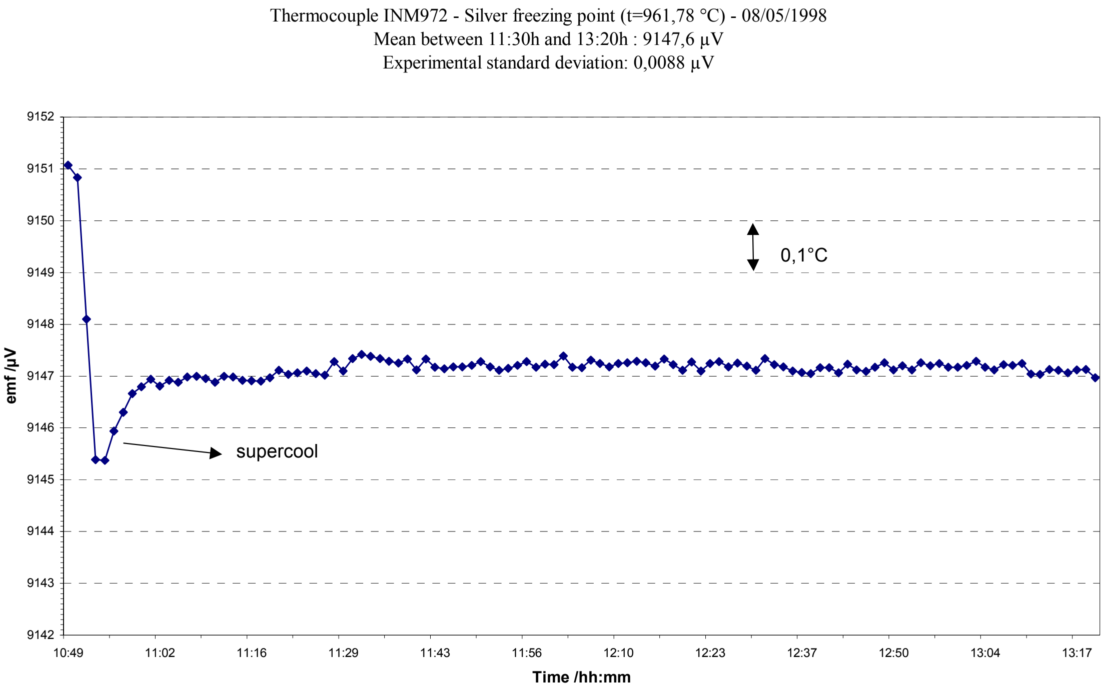
National Metrology Institutes are in charge of supplying traceability for the whole chain of national accredited laboratories in a determined base quantity. The Thermometry Group from Inmetro is responsible for ITS-90 realisation and dissemination in Brazil. Depending on the uncertainties and services which laboratories are accredited, some of them may send noble metal thermocouples or, more recently, pure metal thermocouples, such as Gold-Platinum, Palladium-Platinum, and Gold-Palladium. By analysing their measuring procedures, uncertainties and their facilities during accreditation processes, thermometry group staff started to think about how long information contained in calibration reports of their standards keep on being reproducible. Concerning some changes in calibration conditions, accredited labs may commit some mistakes, such as different depth immersion of standard thermocouple measuring junction during calibration by comparison, inadequate thermal mean evaluation and complete absence of these parameters in their uncertainty budgets. Then, Brazilian Thermometry Group decided to demonstrate the effects caused by inhomogeneity in chemical composition of thermocouple wires.

4. METHODOLOGY FOR INHOMOGENEITY EVALUATING

During the fixed point calibrations, the thermocouples were withdrawn 18 cm from the bottom of the thermometric wells of the cells in order to measure the furnace temperature. In sequence, they were moved down until the bottom with 2 cm steps.

Thermocouple calibrations are realised in these respective fixed points: Silver Freezing Point (961,78°C), Aluminium Freezing Point (660,323°C),. Zinc Freezing Point (429,527°C) and Tin Freezing Point (231,928°C). Graph 1 presents an example of Silver Freezing Point plateau for the type S standard thermocouple INM-972 (Inmetro).

Cold junctions are inserted in Dewar vases which are filled up with a mixture of ice and distilled water. Pre-heated measuring junctions are introduced, alternately, in the wells of fixed point cells, since it is known, by means of the standard thermocouple, that the plateau is set. Extension wires are connected to the voltmeter and the data acquisition program is turned on.

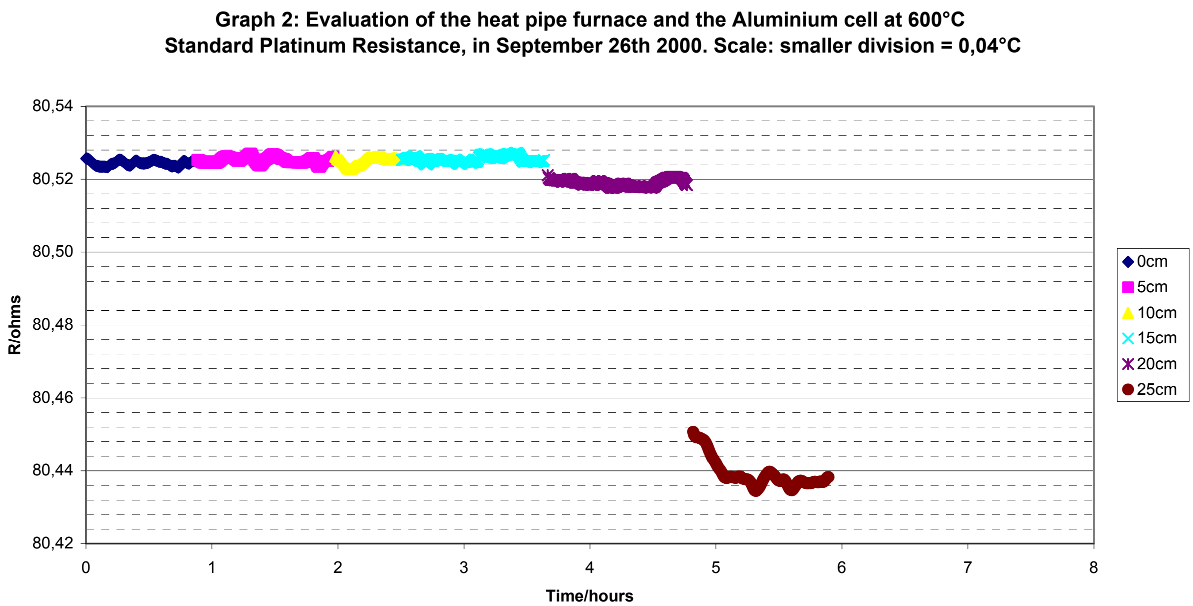


Graph 1 – Silver Freezing Point(Ag 023) for the type S standard thermocouple INM-972.

First, the high temperature furnace had to be evaluated in relation to its temperature uniformity. An aluminium fixed point cell was used as an equalisation temperature block. The furnace controller was set to 600°C, bellow aluminium melting point; afterwards, a well known standard platinum resistance thermometer (SPRT) was introduced in the thermometric well. After establishing the temperature in the bottom of the thermometric well (0 cm), the SPRT is withdrawn 25 cm, from 5 to 5 cm, in order to evaluate the furnace temperature gradient.

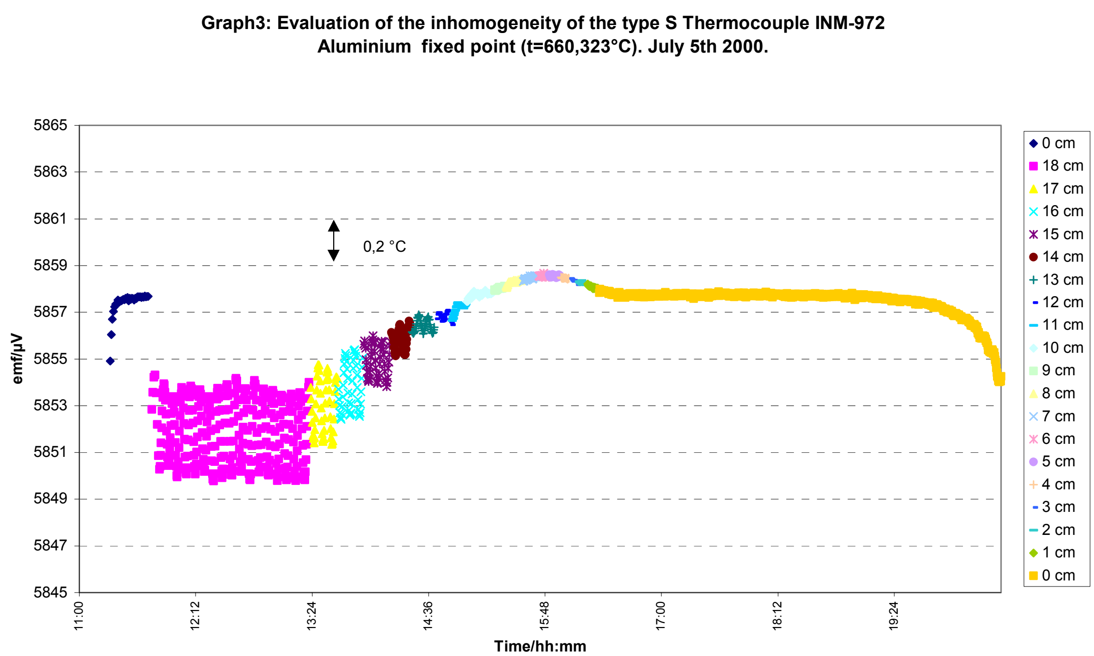
The graphite crucibles of the fixed point cells used in this paper have their depth in about 18 cm, from bottom to the top. Thus, in relation to the graph 2, the largest temperature gradient in the heat pipe measured in 20 cm is about 0,04°C. If we think that the typical uncertainty in fixed point calibration for types R and S thermocouples in Inmetro is ± 0,3°C [1] from 0 to 1100°C (k=2), we should remark that thermal gradient of furnace heat pipe is not a dominant factor in the uncertainty budget.

The next step was to carry out Silver and Aluminium freezing points, testing the best known thermocouples of Inmetro, withdrawing them 18 cm from the bottom of thermometric wells.



Graph 2 – Evaluation of the thermal gradient of furnace.

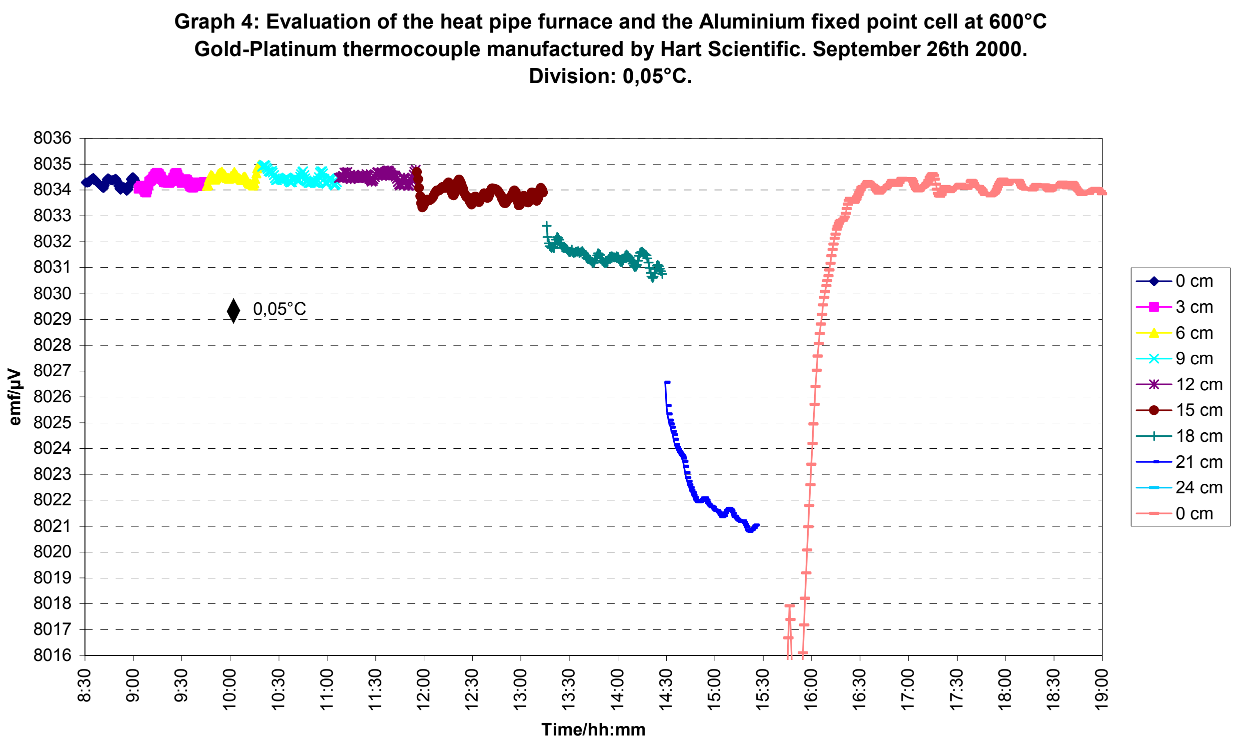
INM-972 is the best known type S standard thermocouple that there is in Thermometry Group.



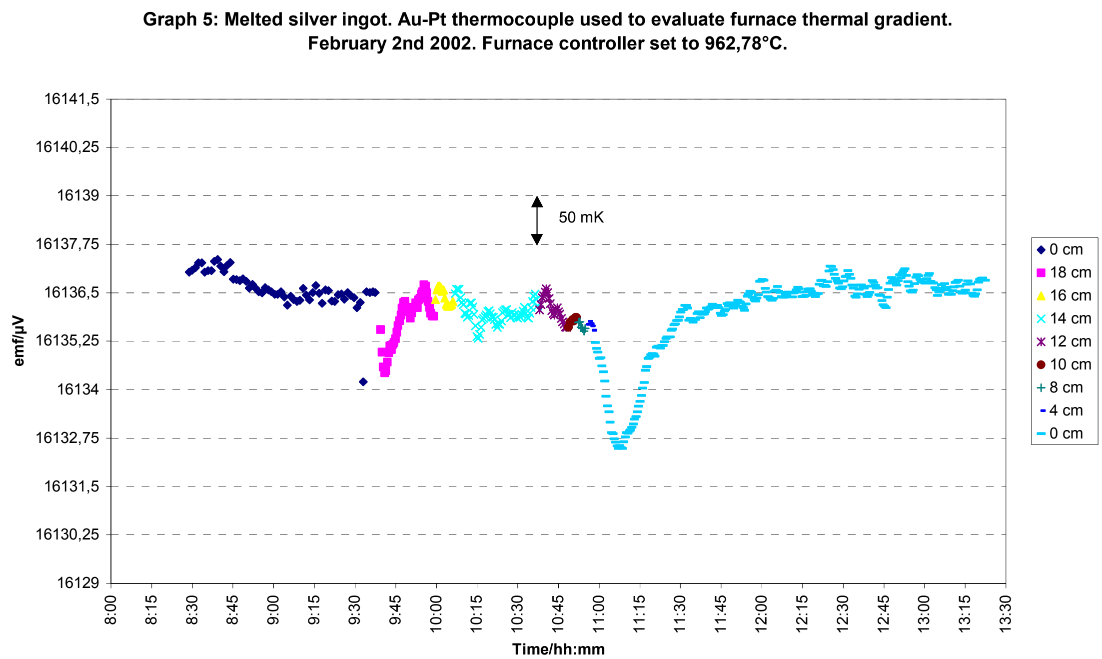
Graph 3 – Evaluation of the inhomogeneity in a type S thermocouple.

As it is shown in graph 3, the fixed point cell seems to have in its crucible a temperature variation of 0,1°C. Yet, the previous test with the SPRT did not show anything similar to that. Then, there were two hypothesis: either there was something quite different with the influence of the furnace in freezing fixed point realisation or inhomogeneity in wires was directly responsible for emf changes. However, the measurements performed in 6 cm and 7 cm depth show the emf above the freezing plateau emfs. Thus, it is possible to demonstrate that is no influence of the furnace temperature in this case, because . the furnace temperature was approximately 0,8°C below the Aluminium Freezing Point (660,323°C).

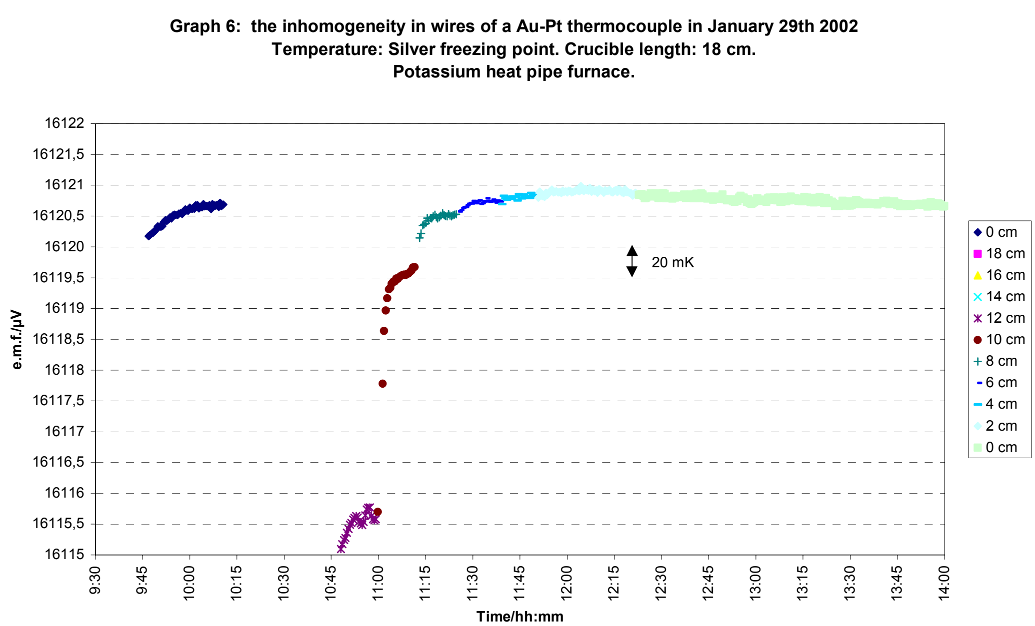
After that, a Gold-Platinum thermocouple was used in order to cease all our doubts. First, as realised with the SPRT, the furnace gradient was tested in 600°C. Afterwards, we carried out both fixed points: silver and aluminium. Bellow graphs bring the information about both tests, though, it is reported only the measurement in silver melting point, because it was the most critical result that was obtained.



Graph 4 – Evaluation of the furnace thermal gradient with Gold-Platinum thermocouple in 600°C.

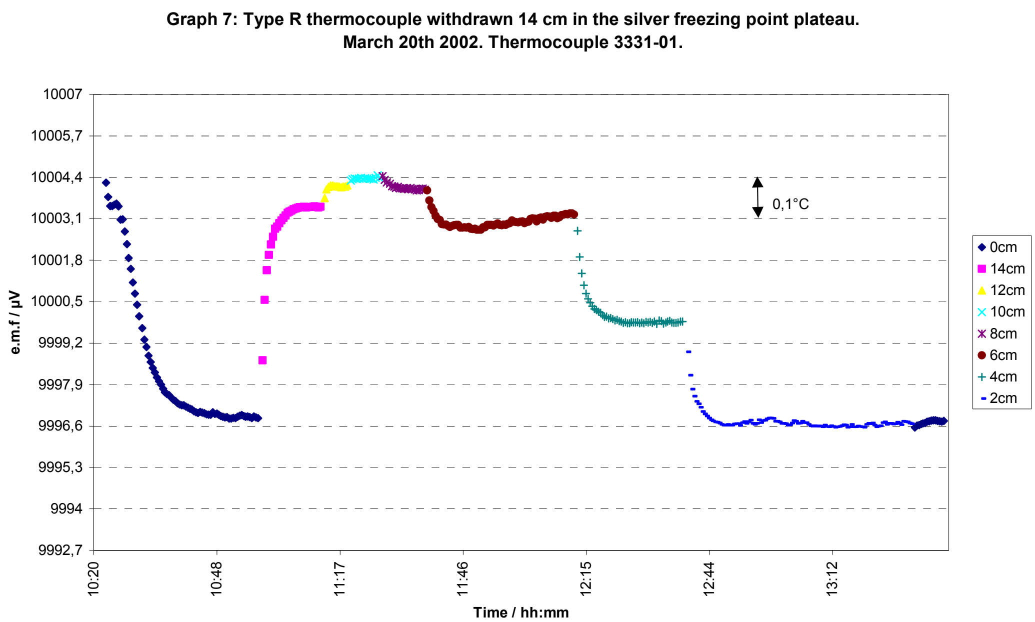


Graph 5 – Evaluation of the furnace thermal gradient with Gold-Platinum thermocouple in 962,78°C.

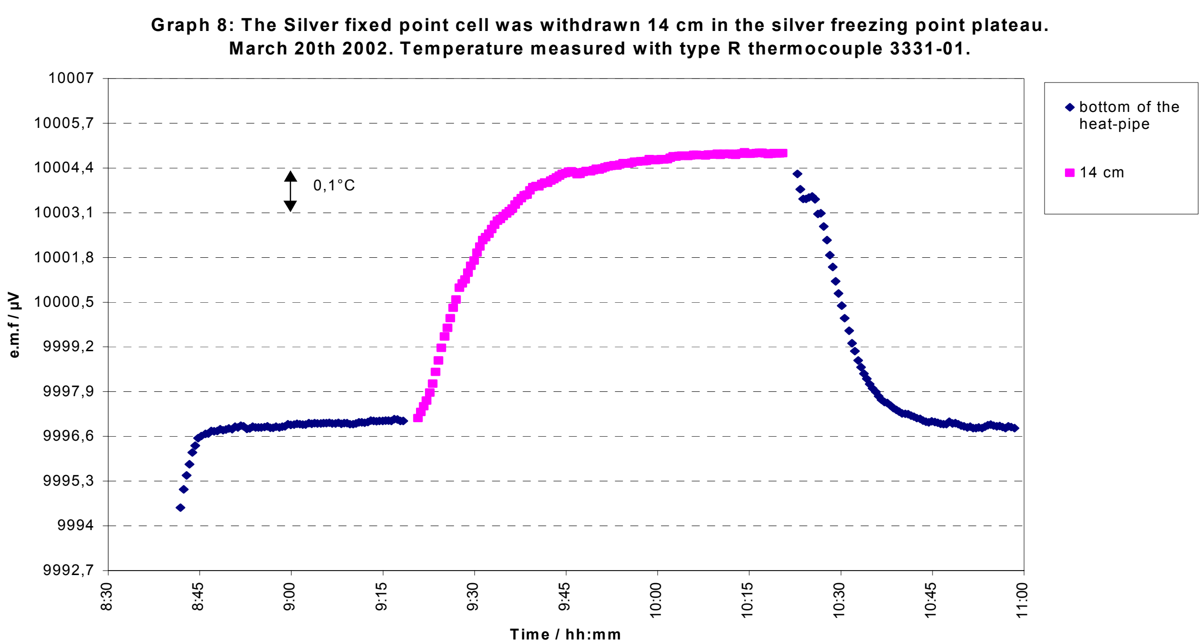


Graph 6 – Evaluation of the inhomogeneity in Silver Freezing point a Gold-Platinum thermocouple.

Type R thermocouple has a behaviour quite similar to the type S. We carried out the same fixed points with a type R thermocouple specially manufactured to this study. This thermocouple has the best alumina sheath and the purest wires that are on disposal in the market. Graphs bellow present results of its inhomogeneity and, as a complementary study, the silver fixed point cell was withdrawn 14 cm from heat-pipe in order to remark that gradient influence throughout wires affects reproducibility even though when we put a kawool fibre cushion beneath the cell.



Graph 7 – Evaluation of the inhomogeneity of a type R thermocouple in the Silver Freezing point.



Graph 8 – Evaluation of the inhomogeneity of a type R thermocouple.

During the plateau of silver freezing point, the cell was withdrawn.

5. CONCLUSION

As it was written at the beginning of this paper, the uncertainty component due to the inhomogeneity is difficult to be assigned in the combined uncertainty [4]. Inhomogeneity effect is represented with the stability component in the thermometry Group uncertainty budget for thermocouple calibrations [1]. We did not succeed in putting them apart, but, with this work, we intend to warn metrologists about the necessity of respecting calibration report information of their standards. They can take for granted that changing depth of the measuring junction of their standard thermocouples, disobeying their calibration protocol and realising calibrations by comparison without a proper study of their furnace thermal gradients, all of these factors are going to prejudice the reproducibility of their measurements.

As it was exemplified in graph 8, the result of a calibration in a shallow furnace may differ from the calibration report supplied by us, because emf generation will be affected by the decrease of measuring junction's insertion into the furnace. Besides, types R and S thermocouples tend to be more sensitive to this depth variation, as seen in graphs 3, 7 and 8.

Gold-platinum thermocouple is more trustful to prevent inhomogeneity effects, as reported in graphs 4, 5 and 6. Thermocouples manufactured with pure wires should represent the future of thermoelectricity .

REFERENCES

1. S.G. Petkovic, F.A.L. Goulart and M.S. Monteiro. FIXED POINT CALIBRATION OF THERMOCOUPLES IN BRAZIL. NCSL International proceedings, 2001.

2. Bedford, R.E., .Bonnier. G., Maas, H. e Pavese, F., TECHNIQUES FOR APPROXIMATING THE INTERNATIONAL TEMPERATURE SCALE OF 1990 - monograph CCT/WG2 - BIPM, 1990, pp.99.

3. G.W. Burns and M. G. Scroger. THE CALIBRATION OF THERMOCOUPLES AND THERMOCOUPLE MATERIALS, 1989, pp. 13.

4. PTB communication, EUROMET workshop in temperature, Paris, 1998.

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